

Clean Harbors Aragonite, LLC  
Aragonite, Utah  
RCRA/TSCA Test Burn Plan  
2007

Test Burn Plan prepared for:  
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APT Project CLA7001

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## Abbreviations and Units

The following abbreviations and units are used in this Test Burn Plan:

Organizations	
APT	Air Pollution Testing
CHA	Clean Harbors Aragonite
EPA	Environmental Protection Agency
STL	Severn Trent Laboratories
UDEQ	Utah Department of Environmental Quality
UDSHW	Utah Division of Solid and Hazardous Waste

Analytes	
Ag	Silver
As	Arsenic
Ba	Barium
Be	Beryllium
Cd	Cadmium
Cl <sub>2</sub>	Chlorine
Co	Cobalt
CO	Carbon Monoxide
CO <sub>2</sub>	Carbon Dioxide
Cr	Chromium
Cu	Copper
HCE	Hexachloroethane
HCl	Hydrogen Chloride
Hg	Mercury
MCB	Monochlorobenzene
Mn	Manganese
Ni	Nickel
NO <sub>x</sub>	Nitrogen Oxides
O <sub>2</sub>	Oxygen
Pb	Lead
PCDDs/PCDFs	Polychlorinated Dibenzo-p-Dioxins and Dibenzofurans
PCB	Polychlorinated Biphenyls
PM	Particulate Matter
POHCs	Principal Organic Hazardous Constituents
Sb	Antimony
Se	Selenium
SO <sub>2</sub>	Sulfur Dioxide
THC	Total Hydrocarbons
Tl	Thallium
(S)VOC	(Semi)Volatile Organic Compound

Units	
@ 7% O <sub>2</sub>	corrected to 7% Oxygen
Btu	British thermal units
C	Celsius
cc	cubic centimeter
cf	cubic foot
cm	cubic meter
ds	dry, standard (preceding a volume – L, cf, etc. – to denote correction to standard conditions of 1 atmosphere, 68°F)
dscfm	dry standard cubic feet per minute
F	Fahrenheit
g	gram
gpm	gallons per minute
Hr	Hour
HRA	Hourly Rolling Average
k, m, u, n, p	kilo, milli, micro, nano, pico (as in liters, grams, etc.)
kcal	kilocalorie
L	Liter
Lb	Pound
M	Molar
min	minute
oz.	ounce
rpm	revolutions per minute
Teq	Toxic equivalents
wt.	Weight

Analytical	
BFB	Bromofluorobenzene
BP	Barometric Pressure
CCV	Continuing Calibration Verification
Cr(C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> ) <sub>3</sub>	Chromic Acetate
Cr <sub>2</sub> O <sub>3</sub>	Chromium Oxide
CVAAS	Cold Vapor Atomic Absorption Spectroscopy
DL	Detection Limit
(HR)GC/MS	(High Resolution) Gas Chromatography, Mass Spectrometry
GFAA	Graphite Furnace Atomic Absorption
H <sub>2</sub> SO <sub>4</sub>	Sulfuric acid
HHV	Higher Heating Value
HNO <sub>3</sub> /H <sub>2</sub> O <sub>2</sub>	Nitric acid/Hydrogen peroxide
HPLC	High Pressure Liquid Chromatography
ICAL	Initial Calibration



ICAP	Inductively-Coupled Argon Plasma Emission Spectroscopy
ICPMS	Inductively-Coupled Argon Plasma Mass Spectroscopy
ICV	Initial Calibration Verification
KMnO <sub>4</sub>	Potassium permanganate
LCS/DCS	Laboratory Control Sample, Duplicate Control Sample
LSC	Laboratory Services Coordinator
LVM	Low Volatile Metal
MDL	Method Detection Limit
MM5	Modified Method 5
MS, MSD	Matrix Spike, Matrix Spike Duplicate
ND	Not Detected
PICs	Products of Incomplete Combustion
QA/QC	Quality Assurance/Quality Control
RB	Run Blank
RCI	Chlorinated organic
RL	Reporting Limit
RPD, RSD	Relative Percent Difference, Relative Standard Deviation
SIM	Selective Ion Monitoring
S/N	Signal-to-Noise ratio
SPCCs	System Performance Check Compounds
SVM	Semi-Volatile Metal
TB	Train Blank
TBP	Test Burn Protocol
TDL	Target Detection Limit
TX/C	Tenax/Charcoal
VOA	Volatile Organic Analysis
VOST	Volatile Organics Sampling Train

Others	
ABC	Afterburner Chamber
ASTM	American Society of Testing and Materials
CEMS	Continuous Emission Monitoring System
CFR	Code of Federal Regulations
CPT	Comprehensive Performance Test
DI	Deionized
DOT	Department of Transportation
HWC	Hazardous Waste Combustor
IATA	International Air Transport Association
ID	Inside Diameter or Identification
LDR	Land Disposal Restrictions
MACT	Maximum Achievable Control Technology
NIST	National Institute of Standards and Technology
ND	Not Detected



PI	Plant Information
PVC	Polyvinyl Chloride
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
RCRA	Resource Conservation and Recovery Act
TSCA	Toxic Substance Control Act
WAP	Waste Analysis Plan

## **1.0 Test Protocol Overview**

### **1.1 Introduction**

[40 CFR §63.1207(f)(v-vii)]

#### 1.1.1 Objectives

Clean Harbors Aragonite L.L.C. (CHA) operates a commercial hazardous waste incinerator in Aragonite, Utah under a RCRA permit issued by the Utah Department of Environmental Quality (UDEQ), Division of Solid and Hazardous Waste (UDSHW); an Air Order issued by the UDEQ, Division of Air Quality (DAQ); and a Title V Permit issued by DAQ. The incinerator operates in compliance with the National Emission Standards for Hazardous Air Pollutants from Hazardous Waste Combustors (40 CFR 63 Subpart EEE). A TSCA coordinated agreement delegates supervision of Polychlorinated biphenyl (PCB) containing waste management to UDSHW and Attachment 17 of the RCRA permit contains PCB waste management requirements. Appendix A contains a Process Description for the CHA incinerator.

CHA is submitting this Test Burn Protocol (TBP) to acquire authorization to conduct a performance test and test burn during calendar year 2007. The purpose for the Test Burn is to:

- Confirm permit compliance with the Air and RCRA permit emission limits.
- Demonstrate that emission requirements are met when the baghouse inlet temperature is 425°F. Operation at 425°F would avoid corrosion of the baghouse structure and bag fabric attack seen while operating at 375°F.
- Obtain test information that can be used as data in lieu of for the Comprehensive Performance Test required in the National Emission Standards for Hazardous Air Pollutants from Hazardous Waste Combustors (40 CFR 63 Subpart EEE) for the final standard described in 40 CFR §63.1219.

These objectives are discussed in further detail below.

#### 1.1.2 Discussion of Objectives

##### 1.1.2.1 Performance Test

The testing conducted during the first test condition will be used to satisfy permit requirements for a performance test during 2007. The applicable permit requirements are as follows:

- State RCRA Permit Condition 5.G.1;
- State RCRA Permit, Attachment 17 Delegated TSCA Approval, Condition 2 (iv)(g); and
- Utah Air Approval Order, Condition 12 Testing Schedule.
- Title V Permit, Conditions II.B.2.a.1(a), II.B.2.b.1(a), II.B.2.c.1(a), II.B.2.d.1(a), II.B.2.e.1(a), II.B.2.f.1(a), II.B.2.g.1(a), II.B.2.h.1(a), II.B.2.i.1(a), II.B.2.j.1(a), and II.B.2.l.1(a).

Condition II.B.2. f.1(a) concerns testing requirements for 40CFR§61 Subpart E – National Emission Standard for Mercury. Subpart E requires mercury testing be conducted in accordance with 40CFR§61 Appendix B Method 101. This differs from the Ontario Hydro Test Method that will be used to sample mercury during this Test Burn. CHA requests to be allowed to use the Ontario Hydro Method to satisfy the mercury testing requirements of 40 CFR§61 Subpart E.

Both permits contain specific requirements concerning test methods. Unless noted otherwise, these will be followed during this Test Burn. If there is conflict between methods, the methods required by the air permits and Subpart EEE will be used. A brief overview of test methods to be used is provided in Table 1-1.

It should be noted that the 2007 Test Burn goals/proposed permit limits represent target values that may or may not be attained during actual testing. Should results less stringent than the present permit be demonstrated, it is expected that permit limits would be changed.

#### 1.1.2.2 Modifications to the Combustion Gas Treatment System

The data from the second test condition will be used to develop data to support modification of the existing RCRA, and/or Air permits to allow the facility to use a modified combustion gas treatment system.

CHA would like to increase the baghouse inlet temperature in order to avoid the attack of the bag fabric and corrosion of the metal surfaces inside the baghouse. The current maximum temperature of 375°F is very close to the acid gas dewpoint and, in some situations, below it. The bag fabric is attacked catastrophically when condensation occurs. Baghouse steel is also badly corroded and must be replaced on a frequent basis.

Subpart EEE requires PCDDs/PCDFs emission below 0.2 ng Teq/dscm @ 7% O<sub>2</sub> for baghouse inlet temperatures above 400°F and 0.4 ng Teq/dscm @ 7% O<sub>2</sub> for baghouse inlet temperatures below 400°F. Data gathered during the formulation of Subpart EEE indicates that more dioxin will be formed at higher

baghouse inlet temperatures. Carbon injection is presently used to control dioxin emission. The carbon rate may be increased for the high temperature condition.

CHA controls mercury by injecting activated carbon and by adding TMT-15© to the scrubber brine. The carbon supplier indicates that mercury adsorption by carbon will be less at increased temperature. CHA may increase carbon feed and may increase TMT-15© feedrate to control mercury emissions in order to meet the Subpart EEE emission limit for the high temperature test condition.

Lead, chromium, and mercury will be added to the combustion chamber feed streams during testing in order to establish allowable metal feed rates. It is planned to extrapolate to determine allowable metal feed rates using the method described in 40 CFR 63.1209 I(1)(v), and (n)(vii) Subpart EEE.

Subpart EEE requires that normal or higher levels of chlorine in waste feed be fed during the comprehensive performance tests (40 CFR 63.1207(g)(1)(A)). During Test Condition 2, chlorine in waste feed will be fed at or near the maximum operating rate of 1496 lbs/hr given in Table 1-3. The treated off gas will also be sampled for chlorine and HCl.

#### 1.1.2.3 HWC MACT Standards

The data from both test conditions will be used as data-in-lieu-of for the Comprehensive Performance Test required to document compliance with the final standard required by the National Emission Standards for Hazardous Air Pollutants from Hazardous Waste Combustors (40 CFR 63 Subpart EEE) in 40 CFR §63.1219.

EPA has promulgated requirements for hazardous waste combustors (HWC), including incinerators, cement kilns, and light weight aggregate kilns that burn hazardous wastes, at 40 CFR 63, Subpart EEE, the HWC maximum achievable control technology (MACT) rule. While the 2007 test burn is not intended to be a Comprehensive Performance Test (CPT) under MACT, many of the MACT requirements will be demonstrated during this test program. CHA plans to use data from this Test Burn to demonstrate compliance with applicable MACT standards.

The intent is to file data in lieu of to demonstrate compliance with the HWC MACT, per 40 CFR 63.1207(c)(2), for the parameters for which compliance with the HWC MACT was demonstrated during the 2007 Test Burn. The data in lieu of will be submitted along with a CPT Plan as required by the HWC MACT. The CPT Plan will identify the testing necessary to demonstrate compliance for those parameters for which compliance was not demonstrated by data in lieu of.

### 1.1.3 Test Resources

Air Pollution Testing will be responsible for test program coordination and implementation and preparation of the final Test Burn report. CHA personnel will collect waste feed and process samples. The laboratories identified in this plan will perform the designated analyses.

## **1.2 Planned Waste Feed and Operating Conditions**

[40 CFR §63.1207(f)(1)(vi) and (vii)]

### 1.2.1 Test Conditions

The Test Burn will consist of two test conditions with each run in triplicate. Both conditions will use the same waste feed. Condition 1 will be run at a bag house inlet temperature of 375°F using approximately 25 lb/hr activated carbon feed rate and will demonstrate compliance with RCRA and Air permit emission limits at current operating limits. Condition 2 will be run at a baghouse inlet temperature of 425°F and carbon and TMT-15 feed rates determined by preliminary tests.

### 1.2.2 Proposed Operating Conditions for the First Test Condition

The first test condition is a performance test to demonstrate compliance with the emission requirements of the RCRA and Air permits. The results of the first test condition will also be used as data-in-lieu-of for the Comprehensive Performance Test required for the final Subpart EEE standard. A performance test can be run at typical operating conditions with emission testing to verify compliance. The data-in-lieu-of is used to establish operating limits and needs to be run at the operating limits that are being established. Accordingly, operations during the first test condition will be run at or near the operating limits required by Subpart EEE and within the operating limits required by the DSHW permit and other air permits.

Table 1-2 lists the target operating conditions for the first test condition.

During the first test condition, stack gas will be sampled at the scrubber exit and/or the incinerator stack. Stack emissions will be measured to determine:

- Destruction and Removal Efficiency (DRE) for principal organic hazardous constituents (POHCs),
- Hydrogen Chloride (HCl) and chlorine (Cl<sub>2</sub>),
- Particulate matter (PM),

- MACT metals (Hg, Pb, Cd, As, Be, Cr), Se, Sb, Ba, Co, Cu, Mn, Ni, Ag, and Tl,
- Polychlorinated biphenyls (PCBs),
- Polychlorinated dibenzo-p-dioxins and dibenzofurans (PCDDs/PCDFs),
- Standard method list of volatile organic compounds,
- Standard method list of semivolatile organic compounds,
- Oxides of nitrogen (NO<sub>x</sub>), carbon monoxide (CO), carbon dioxide (CO<sub>2</sub>), oxygen (O<sub>2</sub>), total hydrocarbons (THC) and sulfur dioxide (SO<sub>2</sub>) using the facility's continuous emission monitoring system (CEMS).

Mercury will be sampled and analyzed in accordance with the Ontario Hydro Speciated Mercury Test Method (aka EPA Pre3, aka ASTM D6784-02) that gives results for both elemental, oxidized, particulate bound, and total mercury emissions.

Chlorinated organics (RCI), required to be reported by the TSCA regulations, will be determined from the volatile organic and semivolatile organic results.

### 1.2.3 Proposed Operating Conditions for the Second Test Condition

The second test condition is a Test Burn to demonstrate that dioxin/furan, mercury, low volatile metal (LVM), and semivolatile metal (SVM) emission requirements for both the Air and DSHW permits are met when operating at a baghouse inlet temperature higher than the present 375°F limit.

Subpart EEE requires operating limits be established for each emission standard. Many of the operating limit parameters are required by more than one emission standard. Subpart EEE allows these requirements to be demonstrated during different Test Burns or Test Burn test conditions as long as the most stringent limit is used as the operating limit for the incinerator. The emission standards for dioxin and furan (40 CFR §63.1209 (k)), mercury (40 CFR §63.1209 (l)), and SVM and LVMs (40 CFR §63.1209 (n)) include an operating limit for baghouse inlet temperature. The operating limits for these emission standards will be established during the second test condition. The incinerator operating limits for operation at a higher baghouse temperature will be determined by using the most stringent of the limits established by the first and second test conditions.

The operating limits that must be demonstrated during a test burn for these three emission standards are listed below:

Dioxin and furan – baghouse inlet temperature, maximum stack gas flow rate, maximum hazardous waste feedrate, and activated carbon feedrate. The EPA has approved an alternative monitoring request that waives CHA's requirement for establishing a minimum blow down rate contained in 40 CFR §63.1209 (m)(1).

Mercury – mercury feedrate, first stage wet scrubber liquid flow rate, second stage wet scrubber liquid flow rate, activated carbon feedrate. TMT-15 is used at the CHA incinerator and CHA has agreed that TMT-15 feedrate is a mercury operating limit.

SVMs and LVMs – baghouse inlet temperature, feedrate of semivolatile metals, feed rate of low volatile metals, feed rate of low volatile metals in pumpable feed streams, feedrate of chlorine and chloride, stack flow rate. The EPA has approved an alternative monitoring request that waives CHA's requirement for establishing a minimum blow down rate contained in 40 CFR §63.1209 (m)(1).

Accordingly, the second test condition will be run with:

- the decided upon baghouse inlet temperature, activated carbon feedrate, and TMT-15 feedrate, and
- maximum hazardous waste feedrate, stack flow rate, mercury feedrate, total SVM feedrate, total LVM total feedrate, pumpable LVM feedrate, and chlorine and chloride feedrate.

Table 1-3 lists the target operating conditions for the second test condition.

During the second test condition, stack gas will be sampled at the scrubber exit. Stack emissions will be measured to determine:

- Hydrogen Chloride (HCl) and chlorine (Cl<sub>2</sub>),
- Particulate matter (PM),
- MACT metals (Hg, Pb, Cd, As, Be, Cr), Se, Sb, Ba, Co, Cu, Mn, Ni, Ag, and Tl,
- Polychlorinated dibenzo-p-dioxins and dibenzofurans (PCDDs/PCDFs),
- NO<sub>x</sub>, CO, CO<sub>2</sub>, O<sub>2</sub>, THC and SO<sub>2</sub> using the facility's continuous emission monitoring system (CEMS).



Again, mercury will be sampled and analyzed in accordance with the Ontario Hydro Test Method EPA Pre3 that gives results for elemental, oxidized, particulate bound, and total mercury emissions.

### **1.3 Description, Preparation, and Delivery of Test Feeds and Spiking Materials**

#### **1.3.1 Test Burn Feed Materials**

As detailed below, POHCs will be spiked into the wastes during the first test condition to provide sufficient feed rates to demonstrate the required DRE. A DRE of 99.9999% will be demonstrated for polychlorinated biphenyls (PCBs); 99.99% will be demonstrated for monochlorobenzene (MCB) and hexachloroethane (HCE). Metals will be spiked into the wastes during the test to provide the target feed rates of metals. The forms of the metals, use of metals extrapolation and activities for metals spiking are discussed below.

As in previous tests, CHA will burn typical waste during the Test Burn. Waste materials to be used will be selected from the wastes on site and available in the marketplace during the period before the Test Burn, and may include incinerator slag that must be reprocessed. The targeted compositions of waste feed materials for the Test Burn are presented in Table 1-4.

#### **1.3.2 POHC Selection and Addition Methodology**

As prescribed by the RCRA permit, the POHCs for the 2007 Test Burn will be HCE, MCB, and PCBs. These POHCs will be added to waste feed materials (input) and measured in the stack gas (output). The solid POHC, HCE, will be fed with the container feed. MCB will be metered into the incinerator using metering pumps. PCBs will be present in waste feeds at sufficiently high levels to demonstrate 99.9999% DRE.

Table 1-5 provides a summary of relevant properties for the selected POHCs. The following criteria were considered during selection of the Test Burn POHCs:

- **Thermal Stability** - At least one of the POHCs selected is in Class 1 of the Thermal Stability List. This list ranks compounds based on their thermal stability outside of the main combustion zone where both temperatures and local oxygen concentrations may be lower, making it more difficult to destroy the compound. The list divides the compounds into seven classes with Class 1 compounds considered to be the most difficult to burn.
- **Heat of Combustion** - At least one POHC ranking low on the basis of heat of combustion is selected. Wastes and POHCs with relatively low heating value are preferred to maximize the mass throughput of the system. However, the

liquid wastes used as an energy source must have a higher heating value (HHV) sufficiently high to sustain combustion.

- Physical State - At least one POHC is a solid and at least one POHC is a liquid.
- VOST Compound - At least one POHC is sampled using the volatile organics sampling train (VOST) (SW-846 Method 0030) procedure.
- Modified Method 5 (MM5) Compound - At least one POHC is sampled using the MM5 (SW-846 Method 0010) sampling procedure.
- Other - Compounds to be burned must be readily available for purchase. Either CHA has stockpiles of these compounds (e.g., HCE), or has previously found them readily available in the marketplace (e.g., MCB, PCBs). Also, analytical interferences and formation of the compounds as products of incomplete combustion (PICs) have been considered.

Solid HCE will be fed to the incinerator with the containerized waste stream. To accomplish this, approximately 10-pound bags of HCE will be affixed to the tops of containers fed to the incinerator during the Test Burn.

CHA intends to feed the liquid POHCs, MCB and PCBs, with the liquid blend waste. MCB will be fed into the liquid blend waste fed to the rotary kiln using a spiking system. PCBs will be present in the liquid blend waste fed to both the rotary kiln and ABC at sufficient concentrations to demonstrate DRE.

Analyses of MCB and HCE will be based upon supplier's certificate of analysis. Analysis of PCB will be based upon laboratory analysis of the waste feed by the Aragonite laboratory.

The metering system for the liquid POHC, MCB, will consist of a metering pump, and a scale. The scale output will feed to a data logger or alternatively to the incinerator distributed process control system with recording by the data historian. The system will be tied into the blended liquid G header at the front wall of the kiln upstream of the block valve. The system will be shut down in the event of a waste feed cutoff. Waste feed sampling of the blended liquid G header will occur upstream of the MCB injection point.

Sufficient quantities of POHCs must be added to the waste feed streams to ensure that enough mass can be detected in the stack gas sample to enable demonstration of the required DRE. Table 1-6 details the minimum and maximum required POHC additions based on anticipated analytical detection limits and the required DRE. The "minimum" and "maximum" calculations pertain to desired values based on the range for calibration of the analytical instrumentation. Note

that the calculations shown for PCBs assume DREs greater than 99.9999% based on actual results from previous trial burns.

### 1.3.3 Metal Addition Methodology

The target feed rates of lead (surrogate for SVM), chromium (surrogate for LVM), and mercury will be accomplished by spiking metal-containing solids and solutions into the incinerator.

The target feed rate of lead (300 lb/hr) will be spiked as a solid and fed with the containerized waste into the kiln. Pre-weighed packages containing known amounts of PbO (lead oxide) will either be placed in, or fastened upon, the waste containers.

Chromium will be spiked into the kiln at the target feed rate of 110 lb/hr. Approximately 77.0 lb/hr of chromium (70% of 110 lb/hr) will be spiked as a solid, and fed with the containerized waste into the kiln. Pre-weighed packages containing known amounts of  $\text{Cr}_2\text{O}_3$  [chromium(III) oxide, or chromic oxide] will either be placed in or fastened upon the waste containers. Approximately 33 lb/hr of chromium (30% of 110 lb/hr) will be fed into the kiln in a pumpable form as a solution of  $\text{Cr}(\text{C}_2\text{H}_3\text{O}_2)_3$  [chromium(III) acetate, or chromic acetate].

Mercury will be fed into the kiln in a pumpable form as a solution of mercuric acetate ( $\text{C}_4\text{H}_6\text{HgO}_4$ ).

The metering system for the liquid metal solutions will consist of a metering pump and a scale. The scale output will feed to a data logger or alternatively to the incinerator distributed process control system with recording by the data historian. The system will be tied into the blended liquid G header at the front wall of the kiln upstream of the block valve. The system will be shut down in the event of a waste feed cutoff. Waste feed sampling of the blended liquid G header will occur upstream of the mercury acetate injection point.

Samples will be taken from each drum of chromium acetate and analyzed for chromium content. Samples will also be taken from each drum of mercuric acetate and analyzed for mercury content. Analyses of solid metal spiking materials will be based upon the supplier's certificate of analysis.

The rates at which metals will be fed will not be above present permit limits.

### 1.3.4 Chlorine Addition Methodology

If the target rate of chlorine (1500 lb/hr) is not available in waste streams and POHCs, concentrated hydrochloric acid and/or polyvinyl chloride scrap will be purchased and fed to the kiln. If hydrochloric acid is added, an acid tanker or

portable acid tank will be connected to the drum pump station pump and Teflon coated feed line and fed to the kiln through either the sludge or direct burn nozzle. Flow will be measured using the Flowmeter installed in the drum pump system. If polyvinyl chloride scrap is used, a bulk solids tank will be committed to this effort and the bulk solids waste and polyvinyl chloride scrap will be mixed together with a backhoe as they are added to the tank.

#### **1.4 Waste Feed and Process Stream Sampling**

Waste feeds, residue (slag and combined spray dryer/baghouse solids), and stack emissions will be sampled during the Test Burn. CHA personnel will sample the waste feed and process residue streams. Air Pollution Testing will collect all stack gas samples. Section 2.0 of this Test Burn Protocol provides significant details on the streams to be sampled, the laboratories performing the analyses, parameters to be measured, sampling frequency and number of samples to be analyzed, and sampling and analytical methods to be used. All labs to be used for this program will be certified by the Utah Bureau of Laboratory Improvement.

Neither the bulk solids nor the containerized waste will contain capacitors or high concentrations of MCB, HCE, or PCB liquids. They will mostly be soils that may contain the POHCs, or ash with residual POHCs. By ignoring the contribution of the POHCs from these two feed streams, any POHCs present will increase the DRE, thus making the reported DRE more conservative.

Incinerator residue streams including slag, spray dryer solids, and baghouse solids will be sampled and analyzed in accordance with the facility Waste Analysis Plan (WAP) for compliance with Land Disposal Restrictions (LDR) per 40 CFR 268. Spray dryer solids and baghouse solids are collected in separate roll-off boxes. Per the WAP, spray dryer solids and baghouse solids will be sampled separately and then composited for analysis. Although the WAP allows weekly compositing and analysis for some parameters, all parameters will be analyzed on daily composites for the Test Burn. In accordance with LDR, the residues must meet all Universal Treatment Standards prior to being shipped offsite for landfill disposal. The CHA laboratory will be responsible for the analyses of these residue streams.

#### **1.5 Gas Sampling**

##### **1.5.1 Sampling Locations**

Gases will be sampled at either or both of two sampling locations, each located downstream of all emissions control equipment. The first location is the wet scrubber exhaust. This approximately 60.0" inside diameter vertical duct is accessed from a series of stairs. The gas flow is vertically down at this site.

Four test ports are available for the sampling. The second location is the approximately 60.5" inside diameter exhaust stack. A set of four ports is accessed from a series of ladders. The gas flow is vertically up at this location. Section 2.6.3.2 provides additional detail on the proposed sampling locations.

### 1.5.2 Sampling Methods

Table 1-1 lists the sampling methods that will be used for the sampling of stack gases. CHA's continuous emission monitoring system (CEMS) will be used to measure CO, CO<sub>2</sub>, NO<sub>x</sub>, O<sub>2</sub>, SO<sub>2</sub> and THC in the stack gas exhaust stream. As required by facility permits, these instruments will operate under current Relative Accuracy Test Audit, Calibration Gas Audit, and Calibration Error tests.

### 1.5.3 Metals Extrapolation Methodology

Subpart EEE allows extrapolation of mercury feed rates and emission rates as described in 40 CFR §63.1209(l)(v) and n(vii). CHA requests it be allowed to use LVM feed rates and emission rates, SVM feed rates and emission rates, and mercury feed rates and emission rates determined during testing to extrapolate to higher feed rates and emission rates.

An average feed rate of lead, chromium, and mercury in each feed and spiking stream will be measured during each test run and used to calculate an average feed rate for each stream. The average feed rates for each stream will be summed to produce a total lead, chromium, and mercury feed rate for each run.

For each metal, the total run feed rate for that metal will be placed on a graph's x-axis and the average metal emission rate for that metal will be placed on that graph's y-axis. The average total metal feed rates will have units of pounds per hour. The average metal feed rates will have units of µg/dscm @ 7%O<sub>2</sub> (µg/dscm @ 7%O<sub>2</sub> are the units used in the Subpart EEE emission standards). A horizontal line will be added to each graph that will represent the Subpart EEE emission standard. A line will be drawn through the origin and the plotted emission rate values. The line will be extended through the horizontal line representing the Subpart EEE emission standard. The metal feed rate on the x-axis that is associated with the intersection of the line and the Subpart EEE emission standard represents the maximum allowable metal feedrate for the incinerator for that metal based upon extrapolation methodology.

The maximum allowable metal feed rate value based upon the extrapolation methodology presented above will be compared to the existing mercury feed rate values. Present and past operating data will be used to determine the historical normal feed rates that are "warranted considering historical metal feed rate data" as specified in 40 CFR 63.1209(l)(1)(ii)(B) and 40 CFR 63.1209(n)(2)(ii)(B). If the maximum allowable metal feed rate value based upon extrapolation is more than

25% above historical normal metal feed rates, the metal feed rate limit will be set at 25% above historical normal metal feed rates. If the maximum allowable metal feed rate limit obtained from the extrapolation methodology is less than 25% above the historical normal feed rate, the metal feed rate will become the extrapolated mercury feed rate.

The historical metal feed rate limits for the Aragonite incinerator are Total Feed rate for Semivolatile Metals (Pb+Cd) 324 lb/hr; Total Waste feed rate for Volatile Metals (As+Be+Cr) 123 lb/hr; Total Mercury Feedrate 0.36 lb/hr. These are the target operating conditions listed in Table 1-2 Target Operating Conditions for the First Test Condition and Table 1-3 Target Operating Conditions for the Second Test Condition.

### **1.6 Conditioning Time Needed to Reach Steady State**

[40 CFR § 63.1207 (f)(1)(xii)]

Three conditioning times will be used during the CPT.

Waste feed, metal spike fed as solid, HCl, and PVC scrap will be fed to the incinerator for at least one and ½ hours before gas sampling begins. The one and ½ hour period is based upon the residence time calculations made to evaluate the incinerator emergency shutdown procedure. These indicate waste residence time is the longest of the time that it takes a lump of waste to travel through the kiln assuming that the lump does not adhere to the side and the time necessary for a lump of waste to reach a temperature where all of the organic would evaporate. During the emergency shutdown procedure the travel time for a lump is increased to 55 minutes by increasing kiln speed to 0.19 rpm. At the lowest permitted kiln speed of 0.15 rpm this calculation gives a residence time of 70 minutes.

Activated carbon will be fed to the crossover duct between the spray dryer and bag house at the Test Burn feed rate for at least the day prior to the Test Burn while waste is fed. The plant is required by permit to feed carbon at 27.8 lb/hr whenever waste is fed. Carbon needs to be fed well in advance of the Test Burn in order to have the amount of carbon in the filter cake that is coating the filter bags at steady state.

Mercuric acetate and metal spike fed as liquid will be added to the blend stream for at least 15 minutes before gas sampling begins. Since the blend is entering the burner, the mercury should be immediately volatilized and travel through the incinerator and emissions control train with the combustion gas stream. The residence time for the gas stream is on the order of 3 minutes.

## **1.7 Request for 720 Hours Pre-Test Burn Operation**

With this Test Burn Plan, CHA is requesting 720 operating hours of pre-Test Burn operation (shakedown), potentially outside of current permit limits, but within the target operational parameters identified in this plan, to adequately prepare for the Test Burn. These operating hours will be used in a sequence of campaigns rather than one continuous period.

Table 1-7 lists suggested limits for the 720-hour pre-test and test period. The table is based upon the target operating limits for the two test conditions given in Tables 1-2 and 1-3. These higher limits allow the flexibility needed to maintain the target limits during the test run and allow testing at higher baghouse inlet temperatures.

## **1.8 Anticipated Test Schedule**

The 2007 Test Burn entails two test conditions each comprised of three test runs. CHA anticipates that the Test Burn will be performed over a period of approximately four days of testing. The tentative schedule for the overall Test Burn period is presented in Table 1-8, and an estimated daily schedule is presented in Table 1-9. Note that this schedule is based on the performance of one or two runs per day.

As nearly as possible, all gaseous sampling trains for the Test Burn will operate simultaneously. The minimum sampling time for the longest train is 180 minutes, but considering port changes, coordination of all sampling activities, and contingency, four to six hours are allowed for each sampling run.

Every attempt will be made to follow the anticipated schedule, although some delays are inevitable. As a guideline, the latest that a sampling run will be initiated will be 14:00 so that it can be assured that the run will be completed by 18:00. This will allow the samples to be collected and recovered and the sampling team to prepare for the next day's test. This guideline will also be used on interrupted testing, such that interrupted trains will be restarted so as to finish by 18:00.

The system will be lined out with all wastes being fed for one hour before initiating testing. If there is a waste feed interruption, the following guidelines will be followed:

- Sampling will be stopped as quickly as possible after the interruption.
- If the interruption is less than five minutes, there will be no line out period, and testing will recommence as soon as possible.

- If the interruption is between five and 20 minutes, there will be a 15-minute line out period, and then sampling will recommence.
- If the interruption is between 20 and 60 minutes, there will be a 30-minute line out period and sampling will recommence.
- If the interruption exceeds 60 minutes, there will be a one-hour line out period.

No test run will proceed without representatives from UDSHW on site unless a written exemption is obtained.

### **1.9 Report**

[40 CFR §63.1207(f)(1)(xxvi)]

The Test Burn results will be submitted to the following agencies within 90 days after completion of the field test program:

Utah Department of Environmental Quality, Division of Air Quality; and

Utah Department of Environmental Quality, Division of Solid and Hazardous Waste (UDSHW).

The report will include summaries of all test results, discussions of any instances where the Test Burn Plan or any specified methods were not followed, determination that the QA and other test objectives were met or a discussion of the impact on the test results, determination of test results, etc.

The Test Burn report will include a detailed discussion of all quality assurance (QA) and quality control (QC) activities conducted in support of the Test Burn. Specifically, the QA/QC section will discuss the conformance (and/or exceptions) with the TBP and any other QA section requirements of methods used during the testing and analysis. Sections of the final report will include:

- Program summary
- Summary of QA/QC Results
- Process Operating Conditions
- Sampling and Analytical Methods Description
- Results of Analysis



- Derived Quantities
- Quality Control and Quality Assurance Program Results Overview

Appendices will provide facility process data, POHC and metal feed rate data, field data sheets, equipment calibration data, CEM calibration data, and analytical data reports. In addition, example calculations will be provided for “derived” quantities. It is planned to submit the appendices in electronic format. The laboratories will be requested to provide data in electronic format to facilitate data review by the regulatory agencies.

A notification of compliance as required by 40 CFR §63.9 (h) will be submitted to The Utah Department of Air Quality with the report.

**Table 1-1 – Overview of Test Parameters and Gaseous Sampling Methods**

Parameter	Sampling Method	Train #
Particulate Matter	EPA Method 5 <b>(a)</b>	1
Hydrogen Chloride and Chlorine	EPA Method 26A <b>(a)</b>	
Semivolatile organics, hexachloroethane and polychlorinated biphenyls	SW-846 Method 0010 <b>(b)</b>	2
PCDDs/PCDFs	Method 0023A <b>(b)</b>	
Total volatile organics	SW-846 Method 0030	3
Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Se, Ag, Tl	EPA Method 29	4
Hg	ASTM D6784-02	5
CO, CO <sub>2</sub> , O <sub>2</sub> , NO <sub>x</sub> , SO <sub>2</sub> , THC	Facility CEMS	N/A
(a) Combined train		
(b) Combined train		

**Table 1-2 – Target Operating Conditions for the First Test Condition**

Operating Limit	Subpart EEE Notice of Compliance July 28, 2005		Current Operating Limit	Test Condition 1 Target Limits
	Reference 40CFR §63.1209	Limit		
Kiln Exit Gas Temperature (°F,HRA)	j(1); k(2)	≥1,815	≥1,820	≥1,820
Afterburner Exit Gas Temperature (°F,HRA)	j(1); k(2)	≥2,018	≥2,018	≥2,018
Spray Dryer Exit Temperature (°F,HRA)	k(1); n(1)	≤375	≤375	≤375
Activated Carbon Feed Rate (lb/hr, HRA)	k(5)(i);l(3)	≥27.8	≥27.8	≥27.8
TMT-15 Feed Rate (lb/hr, HRA)		≥2.36	≥2.36	≥2.36
First Stage Scrubber Feed pH (HRA)	o(3)(iv)	≥5.48	≥5.47	≥5.47
First Stage Scrubber Flow Rate (gpm, HRA)	l(2);o(3)(v)	≥1,928	≥1,960	≥1,920
Second Stage Scrubber Feed pH	o(3)(iv)	≥6.27	≥6.27	≥6.27
Second Stage Scrubber Flow Rate (gpm, HRA)	l(2);o(3)(v)	≥2,141	≥2,141	≥2,141
Total Waste to Kiln (lb/hr, HRA)	j(3);k(4)	≤25,018	≤25,018	≤25,018
Pumpable Waste to Kiln (lb/hr, HRA)	j(3);k(4)	≤7,034	≤7,034	≤7,034
Total Waste to ABC (lb/hr, HRA)	j(3);k(4)	≤10,171	≤10,171	≤10,171
Total Waste Feed of SVMs (lb/hr, 12 Hour RA) (Pb + Cd)	n(2)(ii)	≤338	≤324	≤324
Total Waste Feed of LVMs (lb/hr, 12 Hour RA) (As+Be +Cr)	n(2)(ii)	≤141	≤123	≤123
Total Waste Feed of SVMs (lb/hr, 12 Hour RA) (Pb + Cd)	n(2)(vi)	≤39.1	≤39.1	≤39.1
Total Mercury Feed Rate (lb/hr, 12 Hour RA)	l(1)	≤0.36	≤0.36	≤0.36
Total Chlorine Feed Rate (lb/hr, 12 Hour RA)	n(4); o(1)	≤2,910	≤1,496	≤1,496
HRA – Hourly Rolling Average 12 Hour RA – Rolling Average over 12 Hours				

**Table 1-3 – Target Operating Conditions for the Second Test Condition**

Operating Limit	Subpart EEE Notice of Compliance July 28, 2005		Current Operating Limit	Test Condition 2 Target Limits
	Reference 40CFR §63.1209	Limit		
Kiln Exit Gas Temperature (°F,HRA)	j(1); k(2)	≥1,815	≥1,820	≥1,820
Afterburner Exit Gas Temperature (°F,HRA)	j(1); k(2)	≥2,018	≥2,018	≥2,018
Spray Dryer Exit Temperature (°F,HRA)	k(1); n(1)	≤375	≤375	≤425
Activated Carbon Feed Rate (lb/hr, HRA)	k(5)(i);l(3)	≥27.8	≥27.8	≥40
TMT-15 Feed Rate (lb/hr, HRA)		≥2.36	≥2.36	≥2.36
First Stage Scrubber Feed pH (HRA)	o(3)(iv)	≥5.48	≥5.47	≥5.47
First Stage Scrubber Flow Rate (gpm, HRA)	l(2);o(3)(v)	≥1,928	≥1,960	≥1,960
Second Stage Scrubber Feed pH	o(3)(iv)	≥6.27	≥6.27	≥6.27
Second Stage Scrubber Flow Rate (gpm, HRA)	l(2);o(3)(v)	≥2,141	≥2,141	≥2,141
Total Waste to Kiln (lb/hr, HRA)	j(3);k(4)	≤25,018	≤25,018	≤25,018
Pumpable Waste to Kiln (lb/hr, HRA)	j(3);k(4)	≤7,034	≤7,034	≤7,034
Total Waste to ABC (lb/hr, HRA)	j(3);k(4)	≤10,171	≤10,171	≤10,171
Total Waste Feed of SVMs (lb/hr, 12 Hour RA) (Pb + Cd)	n(2)(ii)	≤338	≤324	≤324
Total Waste Feed of LVMs (lb/hr, 12 Hour RA) (As+Be +Cr)	n(2)(ii)	≤141	≤123	≤123
Total Waste Feed of SVMs (lb/hr, 12 Hour RA) (Pb + Cd)	n(2)(vi)	≤39.1	≤39.1	≤39.1
Total Mercury Feed Rate (lb/hr, 12 Hour RA)	l(1)	≤0.36	≤0.36	≤0.36
Total Chlorine Feed Rate (lb/hr, 12 Hour RA)	n(4); o(1)	≤2,910	≤1,496	≤1,496
HRA – Hourly Rolling Average 12 Hour RA – Rolling Average over 12 Hours				

**Table 1-4 – Target Composition of Test Burn Feed Materials**

Feed Material	Heating Value (Btu/lb)	Chlorine Content (%)	Ash Content (%)
<b>Kiln</b>			
Blended Liquid, A-104	5,000-19,400	2 – 20	0 – 0.5
Direct Burn, A-101	7,000-19,400	2 – 20	0 – 0.5
Aqueous, A-102	0-5,000	0 – 3	0 – 0.5
Bulk Solids <sup>a</sup>	0-7,000	0 – 1	40 – 90
Containers <sup>a</sup>	0-10,000	0 – 20	20 – 90
Sludge, A-103	0-7,000	0 – 2	0 - 10
<b>Afterburner</b>			
Blended Liquid, A-106A/B	5,000-19,400	2 – 20	0 – 0.5
Aqueous, A-105 A/B	0-5,000	0 – 3	0 – 0.5
<sup>a</sup> Containerized and bulk solid waste may include rotary kiln slag that must be reprocessed.			

**Table 1-5 – Relevant Properties for Selected POHCs**

Criteria	HCE	MCB	PCBs
Thermal Stability	Class 5, Rank 209/210	Class 1, Rank 20	N/A
Heat of Combustion Ranking	6	190	39-224 (depends on specific congener)
Heat of Combustion, kcal/g	0.46	6.60	2.31 – 7.75 (depends on specific congener)
Physical State	Solid	Liquid	Liquid
Melting Point, °C	187 (sublimes)	- 45	depends on specific congener
Boiling Point, °C	N/A	132	340 – 375 (depends on specific congener)
Sampling Method	Method 0010	Method 0030	Method 0010

**Table 1-6 – POHC DRE Calculations**

POHC or Analytical Parameter	Sampling Train	Feed Rate (lb/hr)	Assumed DRE	Stack Emissions (g/hr) (d)	Total Collected (e)
MCB - minimum	M 0030	50	99.99%	2.3	670 ng
MCB - maximum	(VOST)	200	99.99%	9.1	2,700 ng
MCB - minimum	M 0030	50	99.9999%	0.023	6.7 ng
MCB - maximum	(VOST)	200	99.9999%	0.091	27 ng
HCE – minimum	M 0010	500	99.9999%	0.23	11 µg
HCE - maximum		2,000	99.9999%	0.91	43 µg
Total PCBs - minimum	M 0010	100	99.99999%	0.005	0.22 µg
Total PCBs - maximum		400	99.99999%	0.018	0.87 µg
Total PCBs - minimum	M 0010	1,000	99.999999%	0.005	0.22 µg
Total PCBs - maximum		4,000	99.999999%	0.018	0.87 µg
<b>Stack Gas Assumptions:</b>	<b>Value</b>	<b>Units</b>			
MM5 Sample Volume	115	dscf			
VOST Sample Volume	20	dsL			
	0.7063	dscf			
Stack Gas Flowrate	40,000	dscfm	<b>Total</b>		
<b>Analytical Assumptions:</b>	<b>MCB</b>	<b>HCE</b>	<b>PCBs</b>		
Reporting Limit	30 ng(a)	0.4 µg (b)	0.02 µg (c)		
Upper Calibration Limit	4,000 ng	NA	NA		
<p>(a) – The 30 ng value is the sum of the 10 ng reporting limits for the front tube and back tube which will be analyzed separately plus 10 ng in the condensate (1 ug/L x 40 ml / 4 – one condensate sample per four tube sets). The 10 ng per tube is lower than the standard RL of 25 ng per tube, but will be supported for MCB based on a method detection limit study to be performed specific for this project.</p> <p>(b) – The 0.4 µg reporting limit for HCE will be achieved with SIM analysis. A 4x dilution factor is included for the split train.</p> <p>(c) – A 4x dilution factor is included for the split train.</p> <p>(d) – g/hr = [feed rate (lb/hr)] x [1 – DRE/100] x [453.6 (gm/lb)]</p> <p>(e) – Collected = [emissions (g/hr)] x [sample vol. (dscf)] / {[stack flow (dscfm)] x [60 (min/hr)]}</p>					

**Table 1-7 – Requested 720-hr Pre-test and Test Run Limits**

Operating Limit	Subpart EEE Notice of Compliance July 28, 2005		Current Operating Limit	Requested 720-hr Pre- test and Test Run Limits
	Reference 40CFR §63.1209	Limit		
Kiln Exit Gas Temperature (°F,HRA)	j(1); k(2)	≥1,815	≥1,820	≥1,750
Afterburner Exit Gas Temperature (°F,HRA)	j(1); k(2)	≥2,018	≥2,018	≥2,018
Spray Dryer Exit Temperature (°F,HRA)	k(1); n(1)	≤375	≤375	≤475
Activated Carbon Feed Rate (lb/hr, HRA)	k(5)(i);l(3)	≥27.8	≥27.8	≥20
TMT-15 Feed Rate (lb/hr, HRA)		≥2.36	≥2.36	≥2.0
First Stage Scrubber Feed pH (HRA)	o(3)(iv)	≥5.48	≥5.47	≥5.0
First Stage Scrubber Flow Rate (gpm, HRA)	l(2);o(3)(v)	≥1,928	≥1,960	≥1,920
Second Stage Scrubber Feed pH	o(3)(iv)	≥6.27	≥6.27	≥6.00
Second Stage Scrubber Flow Rate (gpm, HRA)	l(2);o(3)(v)	≥2,141	≥2,141	≥2, 100
Total Waste to Kiln (lb/hr, HRA)	j(3);k(4)	≤25,018	≤25,018	≤26,000
Pumpable Waste to Kiln (lb/hr, HRA)	j(3);k(4)	≤7,034	≤7,034	≤7,500
Total Waste to ABC (lb/hr, HRA)	j(3);k(4)	≤10,171	≤10,171	≤10,500
Total Waste Feed of SVMs (lb/hr, 12 Hour RA) (Pb + Cd)	n(2)(ii)	≤338	≤324	≤324
Total Waste Feed of LVMs (lb/hr, 12 Hour RA) (As+Be +Cr)	n(2)(ii)	≤141	≤123	≤123
Total Waste Feed of SVMs (lb/hr, 12 Hour RA) (Pb + Cd)	n(2)(vi)	≤39.1	≤39.1	≤39.1
Total Mercury Feed Rate (lb/hr, 12 Hour RA)	l(1)	≤0.36	≤0.36	≤0.36
Total Chlorine Feed Rate (lb/hr, 12 Hour RA)	n(4); o(1)	≤2,910	≤1,496	≤1,500
HRA – Hourly Rolling Average 12 Hour RA – 12 Hour Rolling Average				

**Table 1-8 – Anticipated Test Burn Test Schedule**

<b>Activity</b>	<b>Schedule</b>
Mobilization, site safety training, equipment set-up, preliminary traverses, planning meetings, collection of any field blank trains	Day 1
Conduct Run 1, Run 2	Day 2
Conduct Run 3, any necessary replacement runs, change conditions	Day 3
Conduct Run 4, Run 5	Day 4
Conduct Run 6, any necessary replacement runs	Day 5
prepare samples for shipment, equipment demobilization, ship samples, depart site	Day 6

**Table 1-9 – Anticipated Daily Schedule**

<b>Test Activity</b>	<b>Time</b>
Incinerator lined out on Test Burn wastes	07:00
Begin organic or metal spiking as applicable	08:00
Initiate all stack sampling and waste sampling	09:00
First sampling run	09:00-13:00
Second sampling run	13:00-17:00
Recovery of sampling trains and setting up for next day of testing	17:00 - 19:00
Depart Site	19:00



## **2.0 Sampling and Analytical Program Quality Assurance/Quality Control**

This section presents the Quality Assurance and Quality Control goals, objectives, and procedures for the Clean Harbors Aragonite Test Burn program. The quality assurance/quality control procedures and criteria for this program will comply with the requirements of this document and its updates. The analytical work conducted will incorporate the QA/QC requirements of the approved methods. This document has been prepared using available guidance provided in the following EPA documents:

- “EPA Requirements for Quality Assurance Project Plans,” EPA QA/R-5, November 1999.
- “Component 2 - How to Review a Quality Assurance Project Plan (including Attachment A - Generic Test Burn QAPP,” Hazardous Waste Combustion Unit Permitting Manual, U.S. EPA Region 6, January 1998.
- “Handbook – Quality Assurance/Quality Control (QA/QC) Procedures for Hazardous Waste Incineration” (EPA/625/6-89/023 January 1990).

Quality Assurance Project Plan for Clean Harbors’ Test Burn Program

Facility ID Number: UTD 981 552 177

Prepared for: Clean Harbors Aragonite, L.L.C.

Prepared by: Air Pollution Testing, Arvada, CO 80002

Revision No.: A

Date: March 5, 2007

## **2.1 Title Page**

### 2.1.1 Project Title

Quality Assurance Project Plan for the Clean Harbors Aragonite Test Burn Program

### 2.1.2 Expected Test Burn Date

3<sup>rd</sup> or 4<sup>th</sup> Quarter 2007

### 2.1.3 Project Approvals

\_\_\_\_\_  
Clean Harbors Aragonite Laboratory Analytical Manager

\_\_\_\_\_  
Date

\_\_\_\_\_  
Clean Harbors Project Manager

\_\_\_\_\_  
Date

\_\_\_\_\_  
APT Project Manager

\_\_\_\_\_  
Date

\_\_\_\_\_  
APT Technical Director

\_\_\_\_\_  
Date

\_\_\_\_\_  
STL Knoxville Laboratory Coordinator

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Date

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STL West Sacramento Laboratory Coordinator

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Date

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Focus QA/QC Reviewer

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Date

## **2.2 Table of Contents**

A complete table of contents, including listings of tables and figures and acronyms is presented at the beginning of this Test Burn Plan and includes all pertinent information applicable to this section.

## **2.3 Project Description**

This project will consist of a comprehensive sampling and analysis program designed to demonstrate compliance with current RCRA, TSCA, and Air standards and future HWC MACT rule requirements. Testing will be performed under two process operating conditions, each with triplicate sampling runs. Operating limits for a number of process parameters will be set based on the results of the program. The reader is referred to Section 1.0 for further details on program scope, test objectives and target parameters for emission measurements and process monitoring. Appendix A gives an overall description of the facility. The remainder of this section outlines the detailed measures that will be followed to ensure collection of valid data. A summary of the laboratories performing sample analyses during the program is provided in Table 2-1. A more detailed summary of the sampling and analytical program is provided later in Section 2.6.

## **2.4 Project Organization**

The APT Technical Director, Mr. Paul Ottenstein, will be responsible for the overall direction of this program and will report to the CHA Project Manager, Mr. Karl Libsch. Mr. Ottenstein will be responsible for project design and implementation, communicating with the client, scheduling all activities, review and approval of the Test Burn Plan, reviewing all project data, and preparing all reports. He will be responsible for stack sampling QA/QC. He will be assisted in the oversight of Quality Assurance activities by the APT Project Manager and each Analytical Laboratory Services Coordinator (LSC).

Mr. David Lund, Aragonite Laboratory Supervisor, will be responsible for waste sampling and analysis QA/QC.

Each contract laboratory will have one individual designated as the person responsible for project activities. An independent 3<sup>rd</sup> party QA/QC review of the Test Burn data will be performed by Ms. Terry Bales of Focus Environmental.

### **2.4.1 Project Manager's Responsibilities**

Mr. Alex Mongold will serve as the APT Project Manager and will be responsible for oversight of all on-site sampling and recovery activities. Mr. Mongold will monitor implementation of field and laboratory activities, scheduling performance

and/or system audits as warranted. The Project Manager will report to the CHA Project Manager on any conditions noted which may adversely affect data quality.

Mr. Mongold will be responsible for documenting the collection of all necessary field data forms and field samples, including any audit samples provided by the various regulatory agencies, and field quality assurance samples. Additionally, he will be responsible for ensuring that all samples are transported under appropriate chain of custody to the correct laboratory facilities. He will assist in the preparation of a section for the Final Report summarizing QA/QC activities and will provide an overall evaluation of data quality.

#### 2.4.2 Laboratory Coordinator Responsibilities

Each analytical laboratory will designate a Laboratory Services Coordinator (LSC), who will be the principal point of contact for the APT Project Manager. The LSC will review QA requirements with all laboratory staff to ensure that all required measures are taken to meet data quality objectives. They will monitor the shipment and receipt of samples, track analytical progress, and review data as reported from the laboratories for completeness. Mr. Robert Weidenfeld will serve as the LSC for STL West Sacramento, CA. Mr. Kevin Woodcock will serve as the LSC for STL Knoxville, TN.

Each LSC will be responsible for validation of all data generated by the laboratory for this program and will provide all necessary documentation for inclusion in the final report.

#### 2.4.3. Waste Sampling and Analysis Coordinator Responsibilities

The CHA Laboratory Analytical Manager will be responsible for directing waste feed sampling and analysis and assuring waste feed quality control and assurance goals are met.

### **2.5 QA/QC Program Objectives**

#### 2.5.1 Precision, Accuracy, and Completeness

The collection of data to fully characterize the incinerator waste feed materials and stack gas emissions requires that sampling and analysis procedures be conducted by trained personnel with properly operated and calibrated equipment. QA objectives specific to each analytical methodology performed by the subcontractor laboratories are presented later in Section 2.9. The overall program has been designed with consideration of sampling parameters and analytical limits to ensure that the achieved detection levels for emissions will be

more than adequate for regulatory limit decisions. Detection limits for the various analytes and methods are provided in Appendix B.

Precision is defined as a measurement of mutual agreement among individual measurements made under prescribed similar conditions. Precision is expressed in terms of relative percent difference (RPD) between duplicate determinations (less than 4) and in terms of relative standard deviation (RSD) when 4 or more determinations are made. Overall precision for analysis of the waste feed streams will be assessed through the analysis of one set of duplicate samples for each designated parameter. Precision for the various stack gas samples will be assessed according to the target criteria presented in Tables 2-10 through 2-15.

Accuracy is the degree of agreement of a measurement with an accepted reference or true value. Analytical accuracy will be measured through the recoveries of surrogate spikes, matrix spikes (MS), analysis of standard reference materials, or audit sample analysis. Surrogates are compounds added to samples submitted for organic analyses prior to extraction and analysis; their recoveries are measured to assess sample-specific analytical efficiency and accuracy. Matrix spike samples for the waste feed will be prepared by spiking known amounts of target analytes into a portion of the sample. Matrix spike samples for the stack organic analyses will be prepared by spiking known amounts of target analytes into the sampling media and then carrying the spiked sample through the entire preparation and analysis sequence. Recoveries are monitored to assess laboratory and method accuracy. Laboratory control samples (LCS) will also be used to distinguish between method performance and matrix effects on accuracy. LCS and MS solutions will be independent from calibration standards.

Completeness is a measure of the amount of valid data obtained compared to the amount that was expected under normal conditions. The overall program objective is to obtain valid data for three (3) runs for each test condition. For all data considered critical to the investigation, a completeness objective of 100% has been established. As a result, critical priority data from each of three (3) runs should achieve the precision and accuracy goals established herein. This completeness criterion applies to all permit parameters in emissions samples as well as feed/process stream samples. Individual samples for which the critical data points do not achieve accuracy and/or precision data quality objectives may require reanalysis. Results for samples where matrix interferences preclude meeting objectives for the recoveries of surrogates or spikes will be evaluated for potential bias in calculated emission results. In summary, the completeness goals are stated at 100%, since three valid runs are necessary to assess operation at any one condition.

The possibility always exists that a sample(s) may be lost or broken, and that the data from each individual analytical parameter may not be 100 percent complete

for all test runs. Field blanks, reagent blanks, and archive samples have been incorporated into the sampling and analysis program design in an effort to ensure complete test data and the means to assess overall data quality. The impact(s) of any occurrence of sample loss or failure to meet data quality objectives (DQOs) will be assessed with regards to the specific test objective and/or overall objective of obtaining valid test runs, and will be discussed in the final test report. The completeness objective of the test program is to generate sufficient data for the regulatory agency to judge the performance of the system.

#### 2.5.1.1. Reporting of Analytical Results for Analytes Not Detected

Due to the effectiveness of the facility control equipment, it is anticipated that many target analytes will not be present in the emission samples at detectable or reliably quantifiable levels. Analytical results may be “not detected” (ND), meaning that the amount of analyte was less than the Method Detection Limit (MDL – the minimum concentration that can be measured and reported with a 99% confidence that the analyte is greater than zero) or Target Detection Limit (TDL – one half of the lowest calibration standard), or detected at levels below the RL (reporting limit – the lowest calibration standard level) but above the MDL or TDL. Analytical results presented between the MDL or TDL and RL are appropriately flagged in the analytical reports. In accordance with previous test programs at the Aragonite facility, and consistent with EPA guidelines, the following procedures will be used for reporting of analytical results for analytes not detected or flagged as below the RL in the emission samples.

- As directed in the EPA Handbook “Quality Assurance / Quality Control (QA/QC) Procedures for Hazardous Waste Incineration”, RL values will be used for DRE analytes (PCB, HCE, MCB). This data treatment provides the most conservative value for DRE calculations.
- For dioxin and furan results, the RL values will be used.
- For organic compounds from the Method 0010 and 0030 sampling, metals, HCl and Cl<sub>2</sub>, the RL values will be used for not detected analytes. Flagged values below the RL will be reported as RL values. This approach will provide a conservative emission level for not detected analytes.

#### 2.5.2 Representativeness and Comparability

It is recognized that the usefulness of the data is also contingent upon meeting the criteria for representativeness and comparability. Wherever possible, reference methods and standard sampling procedures will be used. The QA objective is that all measurements be representative of the matrix and operation being evaluated. The detailed requirements for sampling given in the various EPA Reference Methods will be followed to ensure representative sampling of

flue gases. The frequent grab sampling of incinerator feed and process streams during each test run will provide representative samples of these matrices.

The corresponding QA objective is that all data resulting from sampling and analysis be comparable with other representative measurements made by the CPT test team, on this or a similar process operating under similar conditions. The use of published sampling and analytical methods and standard reporting units will aid in ensuring the comparability of the data.

## **2.6 Sampling and Monitoring Procedures**

This section describes the procedures that will be followed during the field sampling program. Throughout the overall program, all sampling will be performed using sampling protocols described herein and approved by EPA. Regulatory agency approval will be obtained for any deviations from, or changes to, the approved TBP which may be warranted prior to program implementation as a result of changes in personnel or facility circumstances. If situations occur during the demonstration testing which necessitate deviations from the plan, the agency will be notified and onsite approval requested. Any deviations from the specified protocols will be fully documented in the final Test Burn Report.

### **2.6.1 Field Program Description**

A detailed description of the compliance strategy and test conditions were provided previously in Section 1.0. In general, however, the program is presently configured to collect samples during three runs under two (2) process operating conditions. Tables 2-3 and 2-4 provide detailed listings of the sampling and analytical parameters and methods planned for this program.

### **2.6.2 Pre-sampling Activities**

Pre-sampling activities include equipment calibration, sample media preparation, cleaning of sample train glassware, preparation of computer-generated sample labels, and other miscellaneous tasks. Each of these activities are described or referenced in the following subsections. Other pre-sampling activities include such details as team meetings, equipment packing and shipment, equipment setup, and finalization of all details leading up to the coordinated initiation of the sampling program.

#### **2.6.2.1 Equipment Calibration**

A most important aspect of pre-sampling preparations is the inspection and calibration of all equipment planned to be used for the field effort. Equipment is inspected for proper operation and durability prior to calibration. Calibration of equipment is conducted in accordance with the procedures outlined in the EPA

document entitled "Quality Assurance Handbook for Air Pollution Measurement Systems; Volume III—Stationary Source Specific Methods" (EPA-600/4-77-027b). Equipment calibration is performed in accordance with EPA guidelines and/or manufacturer's recommendations. Documentation of all calibration records will be kept in the project file during the field program and will be available for inspection by test observers. The final test report will include all pertinent calibration data. Examples of field equipment used and typical calibration requirements follow:

- Probe nozzles - make three measurements of the nozzle ID (to the nearest 0.001 in.) using different diameters with a micrometer. Difference between the high and low values should not exceed 0.004 in. Post-test check - inspect for damage.
- Pitot tubes - measured for appropriate spacing and dimensions or calibrate in a wind tunnel. Rejection criteria given on the calibration sheet. Post-test check - inspect for damage.
- Thermocouples - verify against an electronic calibrator (which is annually certified) at three points (approximately ambient, approximately 250 °F, anticipated stack temperature). Acceptance limits - impinger  $\pm 2$  °F; dry gas meter  $\pm 5.4$  °F; stack  $\pm 1.5$  percent of stack temperature.
- Dry gas meters - calibrate against a reference meter. Acceptance criteria - pretest meter calibration coefficient  $Y_i = 1.00 \pm 0.02$ , post test meter calibration coefficient  $Y_f = \pm 0.05 Y_i$ .
- Field barometer - compare against a mercury-in-glass barometer or use Airport Station BP and correct for elevation. Acceptance criteria -  $\pm 0.1$  in. Hg; post-test check - same.

#### 2.6.2.2 Glassware Preparation

Sample train glassware and sample containers require specialized pre-cleaning to avoid contamination of the sample from the collection container or devices. Cleaning/storage procedures for sample train glassware are summarized below. Note that all bottle caps are fitted with teflon liners which are cleaned in the same manner as the bottles themselves. Sample containers used for waste feed streams are purchased pre-cleaned and sealed to specified EPA protocols.

- EPA Method 0023A/0010 glassware and containers (PCDDs/PCDFs, PCBs and other semivolatile organics (SVOCs)) - wash with soap and water, rinse three times with deionized (DI) water, bake at 400°C for 2-hours, rinse three times with pesticide grade methylene chloride, rinse three times with pesticide



grade toluene and air dry. Open ends will be sealed prior to shipment to the field with clean aluminum foil.

- EPA Method 29/ASTM D6784-02 glassware and containers (metals) – wash with soap and water, rinse with hot tap water, and rinse three times with reagent water. The glassware is next soaked in a 10% nitric acid solution for a minimum of 4-hours, rinsed three times with reagent water, rinsed a final time with acetone, and air dried. All glassware openings where contamination can occur will be covered until the sampling train is assembled prior to sampling.
- EPA Method 26A glassware and components (particulate matter and HCl/Cl<sub>2</sub>) - wash with soap and water, rinse three times with deionized (DI) water, and air dry. Open ends will be sealed prior to shipment to the field with paraffin.
- EPA Method 0030 glassware and containers (volatile organics (VOCs)) - wash with detergent (Alconox) and hot water, rinse three times with HPLC grade water, and oven dry at 110 °C for 2 hours. Open ends will be sealed prior to shipment to the field with clean aluminum foil.

#### 2.6.2.3 Sample Media Preparation

All reagents will be checked in accordance with APT's existing QC Program to minimize the probability of using contaminated solvents. This includes the use of the proper grade reagents/solvents as specified in the test method, selection of reagents from the same lot, and the collection and analysis of the appropriate blanks. Sampling media will be procured and prepared in accordance with the appropriate test methods as described below:

- Tenax and Tenax/charcoal sorbent traps will be conditioned in accordance with protocols outlined in Methods 0030 and/or 5041A.
- XAD resin is purchased new and packed in specially designed sorbent traps. All glass cleaning and sorbent packing procedures will follow the protocols specified in EPA Methods 0023A and/or 0010.
- Quartz filters used in the Methods 5/26A sampling train are purchased from Pallflex Products Co. with designated technical specifications and efficiency ratings.
- Quartz filters used in the Method 29/D6784-02 sampling train are purchased from Pallflex Products Co. who pre-screen filters for metals content.

#### 2.6.2.4 Other Pre-sampling Activities

Sample team meetings will be held to designate responsibilities to each team member. Assignments will be based on individual experience and relative importance of the assigned task. Other pre-sampling activities in the office will include generation of sample checklists, printing of computer-generated sample labels, and proper packing of all equipment. Equipment will then be transported by freight or truck to the sampling location.

Site setup is the final pre-sampling activity. This task will involve moving the equipment to the vicinity of the sample collection area. A separate office trailer or other suitable onsite facility will be used to serve as a sample train setup and recovery area and sample custody area.

Preliminary tests are conducted at the stack location to verify the absence of cyclonic flow conditions and to determine flue gas moisture, temperature, and velocity. These measurements facilitate determination of nozzle size and sample train operation rates for the isokinetic sampling trains.

#### 2.6.3 Sampling Locations

##### 2.6.3.1 Waste Feed and Process Streams

Waste feed materials and process residual streams will be sampled in accordance with acceptable protocols. Waste feed sampling will occur upstream of any POHC or metal spiking location. Taps in the feed lines will be used to access feed streams. Samples will be collected using methodologies described later in Section 2.6.4.

##### 2.6.3.2 Emissions Sampling Locations

Two locations are proposed for collection of emissions samples for the 2007 Test Burn – the incinerator stack and the scrubber outlet. The emissions ductwork from the scrubber outlet to the incinerator stack is a closed system and emissions are equal at the two locations. When this document refers to “stack” samples, it is understood to mean samples collected at either the incinerator stack or the scrubber outlet. Access to the scrubber outlet location is substantially easier than the incinerator stack, and the available working space is also much greater. Consequently, it is proposed to collect most or all emissions samples at the scrubber outlet.

The incinerator stack is constructed of fiberglass and has a height of 149 feet with an inner diameter that has previously been measured at 60.5 inches. Four sampling ports arranged 90 degrees apart are available for the test program at a height of 58 feet above ground level, and are accessed from a series of ladders.

The stack diameter at the sampling location, as well as distances to upstream and downstream flow disturbances, will be determined on-site prior to emissions sampling. It is expected that twelve (12) test points will be required for isokinetic sampling trains at the stack location.

The scrubber outlet, which is downstream of all operating control equipment, provides an additional four sampling ports which are accessed from stairs. The inner diameter has been previously measured at 60.0 inches at this location. As with the incinerator stack location, all required dimensions will be confirmed on-site prior to sampling. It is expected that sixteen (16) sampling points will be required for isokinetic sampling trains at the scrubber outlet location.

#### 2.6.3.2.1. Additional Notes on the Scrubber Outlet Location

The direction of gas flow at the scrubber outlet is vertically down, and the saturated gas stream has been observed to contain droplets near the stack walls. Historically, this has been dealt with by adjusting the exterior sampling points inward by several inches to avoid over collection of the droplets.

This procedure has been approved by the on-site regulator in previous test programs and is not expected to have any adverse impact on data quality. It is consistent with procedures provided in Method 1 (40 CFR 60 Appendix A) for cases where the outermost calculated sampling point or points are too close to the stack wall.

#### 2.6.4 Waste Feed and Process Stream Sampling Procedures

All waste feed and process stream sampling will be performed by facility personnel. Each sample will be assigned a unique sample code for identification. Sufficient quantity will be collected to allow for sample splits, backup or archived samples, and duplicates, as applicable.

##### 2.6.4.1 Waste Feed

Containerized wastes will be characterized by analysis or application of the Matrix Protocol (as detailed in the facility Waste Analysis Plan - WAP) prior to the materials being repacked into Test Burn feed drums as described below. Bulk solids will be sampled at the apron feeder access port above the bulk solids flop gates. Pumpable sludge will be sampled from a tap in the sludge feed line at the front wall. Kiln aqueous liquid waste will be sampled from a tap in the aqueous feed line at the kiln. ABC aqueous liquid waste will be sampled from taps in the aqueous feed line at the afterburner. Blend liquid to the kiln will be sampled from a tap in the blend liquid line at the front wall. Blend liquid to the ABC will be sampled from taps in the blend liquid line at the ABC. Sludge, aqueous liquid,

and blend liquid to each chamber will be fed to the system from one tank at a time for each stream. Fuel oil will be sampled at the fuel oil storage tank.

Grab samples of the liquid streams will be collected during each run from sample taps. The sample tap is opened and the line is flushed with the material being collected. The flush is then discarded into a container and managed appropriately, then the specified sub-sample is collected. This ensures that the actual material collected is representative of the stream. Liquid is collected into a large beaker or sample jar at the prescribed frequency. Approximately 100-mL of the sample will be transferred into a larger sample bottle. This composite sample will be analyzed for nonvolatile parameters. Liquid samples will be taken every 15 minutes during the test.

Samples of the apron feed solids will be obtained by compositing sub-samples collected at 15-minute intervals. At the prescribed frequency, a 250-mL beaker (or sample jar) will be filled, and the material will be transferred into a larger sample container. The resulting composite will be analyzed for nonvolatile parameters. If volatile analyses are required, a separate 4-oz. sample jar will be filled at the required frequency; these samples will be composited at the laboratory.

The containers to be fed during the Test Burn will be those available at the time of the test. Samples will be taken according to the facility Waste Analysis Plan. POHC content in containerized waste will not be used to calculate DRE.

Analysis and quality control of waste feed samples will be in accord with the Laboratory SOPs used by CHA to analyze waste samples.

Appendix F – Waste Feed, Spiking, and Raw Material Sampling and Analysis Plan provides detail on waste feed sampling.

#### 2.6.4.2 Process Streams

Slag solids will be sampled from the roll-off box below the deslagger. Spray dryer solids will be sampled from the discharge chute. Baghouse solids will be sampled from the sample access port on the screw conveyor in the loadout building. Baghouse solids and spray dryer solids will be composited to yield one sample in accordance with the facility WAP.

#### 2.6.4.3 Spiked Materials

Given that the solid POHC (HCE), liquid POHC (MCB) and solid metal spikes are pure materials of known analysis, no sampling will be conducted. Rather, the purity of these materials will be based on certificates of analysis provided by the vendor. Containers of liquid metal spiking solutions will be sampled to determine

composition. Individual bags of HCE will be weighed prior to placement into or onto a drum.

If PVC scrap is added to the bulk solids stream to increase its chlorine content, a grab sample will be taken from each PVC container and composited. The chlorine analysis for the composite will be used in calculating bulk solids chlorine content.

Should purchased HCl be added to the incinerator in order to increase the amount of chlorine fed, the purity of this material will be based upon the certificate of analysis supplied with the acid shipment.

#### 2.6.5 Stack Sampling Methodologies

Gases discharged from the exhaust stack will be sampled for the following parameters:

- Flue gas velocity, flow rate, temperature, and moisture content;
- MACT metals – As, Be and Cr (LVM); Cd and Pb (SVM); and Hg;
- Other Metals – Se, Sb, Ba, Mn, Ag, Tl, Ni, Co, Cu;
- Particulate matter;
- HCl and Cl<sub>2</sub>;
- Volatile POHC and volatile organics;
- PCDDs/PCDFs;
- PCBs, HCE and SVOCs;
- Carbon Monoxide (CO), oxygen (O<sub>2</sub>), total hydrocarbons (THC), sulfur dioxide (SO<sub>2</sub>), oxides of nitrogen (NO<sub>x</sub>) and carbon dioxide (CO<sub>2</sub>) using the facility's CEMS.

The following sections provide summaries of the sampling methodologies to be followed. In addition, sample field data sheets to be used during the program are provided in Appendix C. Summaries of relevant information pertaining to setup and recovery of each isokinetic sampling train are provided in Appendix D.

##### 2.6.5.1 Gas Stream Velocity, Moisture, and Fixed Gases

Gas stream flowrate, moisture, and fixed gas concentration will be determined concurrent with each of the isokinetic sampling trains. Gas stream velocity will be determined using a pitot tube and water manometer in accordance with EPA Method 2. Gas stream temperature will also be determined at each of the Method 2 traverse points using a Type "K" thermocouple and pyrometer. Gas stream moisture will be determined as specified in EPA Method 4 concurrent with the isokinetic sampling methods. In this procedure the impinger contents are measured or weighed before and after each test run and used in conjunction with the metered gas volume to determine the gas stream moisture content. The facility's stack CEMS will be used for measuring O<sub>2</sub> and CO<sub>2</sub> for gas stream molecular weight determination and constituent oxygen correction at both gaseous sampling locations. The facility CEMS is operating under a current Relative Accuracy Test Audit, and the data are of sufficient quality for the data quality objectives of this Test Burn.

#### 2.6.5.2 Volatile Organic Sampling Train (VOST) for Volatile POHC

EPA Method 0030 will be used to determine stack gas concentrations of the volatile POHC and volatile organics. Data collected from VOST samples will be used to calculate the destruction removal efficiency (DRE) for the volatile POHC, monochlorobenzene. The VOST method utilizes Tenax and Tenax/Charcoal cartridges to adsorb target volatile organic compounds; each cartridge is preceded by a condensing module. Specific sampling details for the Method 0030 train are as follows:

- Sampling rate – 0.5 - 1.0 Lpm (the high moisture content of the stack gas has previously necessitated slower sampling rates due to high sample vacuum – the method requirement of 20 L per tube set will be maintained which will result in longer sampling times for the reduced flow rate)
- VOST pair run time – 20 – 40 minutes (see above)
- VOST tube pairs collected per run - 4 (a, b, c and d)
- VOST tube pairs designated for analysis - 3 (a, b and d)
- Minimum probe temperature - 135 °C
- No. of field blank pairs collected – 3
- No. of trip blank pairs collected - 1

The recovery activities for the VOST method will include:

- Sealing the sorbent cartridges with Swagelok fittings and placing them in their original glass culture tubes with glass wool to absorb shock.
- Transferring the collected condensate into a 40 mL VOA vial, diluting to volume with DI water to eliminate headspace and the possibility of revolatilization of the compounds.
- Further reducing reactivity by storing all samples at 4 °C.

#### 2.6.5.3 Metals

EPA Method 29 will be utilized for the collection of target metals other than mercury. An optional moisture knock-out impinger may be employed due to the expected high moisture levels in the Clean Harbors Aragonite exhaust gas. Target analytes include:

- MACT LVM metals – arsenic, beryllium and chromium;
- MACT SVM metals – cadmium and lead and;
- Other metals – selenium, antimony, barium, manganese, silver, thallium, nickel, cobalt, copper.

Specific sampling details for the Method 29 sampling train are as follows:

- Target sampling rate - 0.75 cfm
- Sample run time – 2 hr
- No. of sampling points per stack traverse – 6 or 8
- Total No. of sampling points – 12 or 16
- No. of field blanks collected – 1
- No. of reagent blanks collected – 1 per batch

#### 2.6.5.4 Mercury

ASTM Method D6784-02 (aka The Ontario Hydro Speciated Mercury Sampling Train, aka Pre 3) will be utilized for the collection of mercury. An optional moisture knock-out impinger may be employed due to the expected high moisture levels in the Clean Harbors Aragonite exhaust gas. Specific sampling details for the ASTM D6784-02 sampling train are as follows:

- Target sampling rate - 0.75 cfm
- Sample run time – 2 hr
- No. of sampling points per stack traverse – 6 or 8
- Total No. of sampling points – 12 or 16
- No. of field blanks collected – 1
- No. of reagent blanks collected – 1 per batch

#### 2.6.5.5 Particulate Matter (PM), HCl, and Cl<sub>2</sub>

Sampling for PM, HCl, and Cl<sub>2</sub> will be performed in accordance with EPA Method 26A. The Method 26A HCl/Cl<sub>2</sub> train will also serve as the Method 5 PM train. An optional moisture knock-out impinger may be employed due to the expected high moisture levels in the Clean Harbors Aragonite exhaust gas. Specific sampling details for the Methods 5 and 26A sampling train are as follows:

- Target sampling rate - 0.75 cfm
- Sample run time – 2 hr
- No. of sampling points per stack traverse – 6 or 8
- Total No. of sampling points – 12 or 16
- No. of field blanks collected – 1
- No. of reagent blanks collected – 1 per batch

#### 2.6.5.6 PCDDs/PCDFs and Other Target Semivolatile Organic Parameters

A combined Method 0023A/0010 sampling train will be used to sample for all target parameters. An optional moisture knock-out impinger may be employed due to the expected high moisture levels in the Clean Harbors Aragonite exhaust gas. PCDDs/PCDFs will be collected following the procedures outlined in EPA Method 0023A. PCBs, HCE, and SVOCs will be collected following the procedures outlined in Method 0010. Sample train recovery procedures will follow Method 0023A, except that the toluene rinses will be recovered in separate containers from the acetone/methylene chloride rinses to allow all appropriate analyses. Specific sampling details for the Method 0023A/0010 sampling train are as follows:



- Target sampling rate - 0.75 cfm
- Sample run time – 3 hr
- Minimum sample volume required (as per Subpart EEE) - 2.5 dscm
- No. of sampling points per stack traverse – 6 or 8
- Total No. of sampling points – 12 or 16
- No. of field blank trains collected – 1
- No. of reagent blanks collected – 1 per batch

#### 2.6.5.7 Continuous Emissions Monitoring (CEM)

CHA will provide continuous emission monitoring for parameters including CO, O<sub>2</sub>, CO<sub>2</sub>, CO, SO<sub>2</sub>, THC, and NO<sub>x</sub> in accordance with existing permit requirements. These data will be collected in accordance with the existing facility CEM QA Plan.

### **2.7 Sample Handling, Traceability, and Holding Times**

Sample integrity will be maintained throughout all phases of the sampling and analysis program. Samples will be held within sight of the samplers or sample custodian, or will be kept in sealed or secured containers at all times. Sealed coolers and DOT shipping boxes will be used to ship samples to the designated laboratory via Priority 1 overnight FedEx service or they will be personally delivered by APT personnel. Shipping will be conducted in strict accordance with applicable DOT and/or IATA regulations.

Preprinted sample identification labels are used by APT to ensure that all required information is fully documented. When sample batches are shipped to the specified laboratory, a Chain-of-Custody form (see Appendix C) accompanies the shipment. This form is based on established laboratory format and will be used to document sample transfer in the field and from sampling personnel to the laboratory.

The APT Project Manager will coordinate the packing and shipment of all samples. Worksheets specifically designed for this program will be generated prior to the field effort. These sheets will assist the Project Manager in assuring that all samples have been collected, accounted for, and shipped under sample traceability documentation to the appropriate laboratory. Requirements pertaining to sample preservation and recommended holding times are noted in Tables 2-5 and 2-6.

All materials such as field and laboratory notebooks and logbooks, field and laboratory data records, correspondence, reports, sample tags, traceability records, and instrument printouts will be clearly labeled with the project number and will become a permanent part of the project file. Project samples will be disposed of in an appropriate manner 60 days after acceptance and approval of a final report. All project-related documentation at both APT and the subcontractor laboratories will be kept on file for 2 years following submittal of the final report.

## **2.8 Analytical Methods and Calibration Procedures**

This section delineates the analytical protocols that will be used to analyze samples during this Test Burn. Samples of waste feed materials, process streams, and stack gas will be collected and analyzed for the parameters previously discussed using the appropriate laboratory protocols detailed in this section and as outlined in Table 2-3 and 2-4.

### **2.8.1 Analysis of Waste Feed and Process Streams**

All waste feed and process stream samples will be analyzed by or under the direction of the onsite Aragonite laboratory. Waste feed materials include containerized solids, apron feed solids, pumpable sludge, direct burn liquids, aqueous waste, and liquid blend. Process streams include kiln slag and residue material from the baghouse and spray dryer.

#### **2.8.1.1 Chemical and Physical Properties of Process Streams**

Analyses to determine the chemical and physical properties of the waste feed materials will be performed using appropriate ASTM or EPA SW-846 analytical methods as outlined in Table 2-2. Quality assurance requirements for the chemical properties of these materials are summarized in Tables 2-7, 2-8, and 2-9.

#### **2.8.1.2 Metals in Waste Feed and Process Streams**

These streams will be analyzed for the following target metals: arsenic, beryllium, cadmium, chromium, lead, mercury, and nickel. Analyses for metals other than mercury will be performed using inductively-coupled argon plasma emission spectroscopy (ICAP) as described in EPA Method 6010B (SW-846, 3rd edition).

Mercury analysis will be performed using EPA Methods 7470A or 7471A (SW-846, 3rd edition). Quality assurance requirements for the analyses of metals in waste feed materials are summarized in Table 2-8. As previously discussed, surrogate inorganic metal feeds in drums or bulk will not be sampled and

analyzed, but rather a certificate of purity will be provided by the supplier/manufacturer of these commercial grade materials.

#### 2.8.1.3 POHCs in Waste Feed Materials

Target POHCs for the program include hexachloroethane, monochlorobenzene, and total PCBs. POHC input rates will be determined as outlined in Table F-1 in Appendix F. Analytical methods for these POHCs include EPA Methods 8260B for MCB, 8270C for HCE, and 8082 for PCBs. Quality assurance requirements for these analyses are summarized in Table 2-9.

#### 2.8.2 Analysis of Stack Gas Samples

##### 2.8.2.1 Stack Gas-VOST Analysis

Stack gas samples will be analyzed for MCB and other volatile organics using EPA Method 5041A (VOST tubes) and EPA Method 8260B (condensate).

Analysis — The samples collected from each VOST run will consist of Tenax cartridges, Tenax/charcoal backup cartridges, and a flue gas condensate. Cartridges will be desorbed and analyzed for volatile organics using the thermal desorption GC/MS procedures specified in Method 5041A of SW-846. Condensate samples will be analyzed using Method 8260B. All VOST tubes from each run will be analyzed separately to confirm that significant analyte breakthrough has not occurred. If the back tube contains < 30% of the amount of the POHC found on the front Tenax trap, or if the back trap contains less than 75 nanograms of the POHC, breakthrough will not be considered to have occurred.

On a case-by-case basis, detected analyte in the back tube may be assessed as indicative of analyte affinity for the charcoal collection media (present only in the backup cartridge) rather than breakthrough; chloromethane has been observed to collect preferentially on the backup tube.

All QA/QC requirements of EPA Method 5041A for instrument calibration and performance will be met prior to sample analyses, including:

- System performance checks using the five system performance check compounds (SPCCS) will be conducted initially and after every 12 hours of analysis. The minimum response factors for the volatile SPCCS will be 0.300, except for chloromethane, bromoform, and 1,1-dichloroethane, where the minimum response factors are 0.100.
- Daily calibration of the system, including evaluation of the internal standard responses and retention times in the check calibration standard. Performance criteria specified in the method will be used to determine whether

the system has malfunctioned. If samples are analyzed under conditions of malfunction, an evaluation of the impact of that malfunction on data quality will be performed, with the results of the investigation presented in the final report.

Thermal desorption will be conducted using a VOST Tube Clamshell desorption unit which is designed to accommodate sorbent cartridges in series. The desorption gas is plumbed to direct flow through each pair of traps, then through a purge vessel to trap desorbed water and, finally, onto the head of a smaller sorbent column (K-trap) which is located in a Tekmar 3000 purge and trap device. The volatile components are then thermally desorbed onto the GC by heating the trap to 250 °C. Prior to analysis, the volatile surrogate compounds listed in the method will be flash vaporized onto each Tenax cartridge set. The internal standards are added to the purge water.

The analytical performance check for the designated POHC will be completed prior to the program in accordance with SW-846 Method 0030, Section 7.1 by the laboratory conducting the analyses. The amount spiked for this analysis will be at or near the anticipated "critical level" for the POHC for this project (approximately 250 ng) and will be recognized as having passed the check if the recovery is within 50% - 150% of the expected value.

Calibration for Method 5041A - The GC/MS will be tuned to BFB (bromofluorobenzene) at the beginning of each 12-hour analysis sequence, applying the acceptance criteria for key ion abundance listed in the method.

Upon compliance with all system criteria, the GC/MS will be initially calibrated at a minimum of five calibration levels using internal standards and surrogates.

Calibration standards for the POHC must cover the range of concern for DRE demonstration. The high concentration standard for each POHC must be at a minimum 800 ng; the lowest calibration standard analyzed must be 200 ng or less. Calibration procedures follow STL Knoxville SOP: KNOX-MS-0011 which is based on methods 0031, 5041A, and 8260B (SOP available upon request).

Response factors versus the internal standard will be calculated for all components at each level of calibration. Verification of a single point of the calibration curve will be performed for each 12 hours of sample analysis. QA/QC requirements for VOST analyses are provided in Table 2-10.

#### 2.8.2.2 Metals in Stack Gas Samples

Analysis - Each sampling train will be prepared and analyzed in accordance with EPA Reference Method 29.

Two samples are generated for analysis from each sampling train. The first sample, labeled Fraction 1A, will be the digested sample from the front half of the train, consisting of the particulate filter, the evaporated residue from the front-half acetone rinse (as applicable), and the front-half nitric acid probe rinse. Fraction 2A consists of digestates from the moisture knock out and HNO<sub>3</sub>/H<sub>2</sub>O<sub>2</sub> impingers 1, 2, and 3.

Analyses for metals will be performed using Inductively Coupled Argon Plasma Mass Spectroscopy (ICP-MS) as described in EPA Method 6020 (SW-846, 3rd Edition). All quality control procedures, including the interference check standard, will be followed as described in the respective method.

Calibration—Calibration of the ICP-MS will be performed daily in accordance with the procedures described in Method 6020 and the manufacturer's instructions. The calibration is verified daily by analysis of an instrument check standard prepared from an EPA quality control concentrate or other independent standard. QA/QC requirements for the analysis of metals in stack gas samples are summarized in Table 2-11.

#### 2.8.2.3 Mercury in Stack Gas Samples

Analysis - Each sample will be collected in accordance with ASTM Method D6784-02 (a.k.a. PRE-3) and analyzed in approximate accordance with EPA Method 7470A (SW-846, 3rd Edition). STL standard operating procedures, which will be made available upon request, will be strictly followed.

Five individual samples are generated for analysis as four sample fractions from each sampling train. Labeled fractions 1 and 2 consist of the front-half, particulate-bound mercury. These fractions are microwave digested and composited prior to analysis. Labeled fraction 3 is the contents of the first three impingers (seeded prior to sampling with 1.0 M potassium chloride), along with appropriate rinses. This fraction is the gaseous Hg<sup>+2</sup> (oxidized) mercury. Labeled fraction 4 is the contents of the fourth impinger (seeded prior to sampling with 5% HNO<sub>3</sub>, 10% H<sub>2</sub>O<sub>2</sub>), along with appropriate rinses. This fraction is part of the elemental mercury. Labeled fraction 5 is the contents of the fifth through seventh impingers (seeded prior to analysis with 4% KMnO<sub>4</sub>, 10% H<sub>2</sub>SO<sub>4</sub>), along with appropriate rinses. This fraction is the remainder of the elemental mercury. Calibration – Calibration of the Cold Vapor Atomic Absorption System will be performed daily, generating a 7-point curve (a blank and six calibration standards). Calibration is checked initially with a second source initial calibration verification (ICV) standard, and on an ongoing basis with continuing calibration verification (CCV) samples. Background contamination is similarly assessed with an initial calibration blank and continuing calibration blank samples.

QA/QC requirements for the analysis of mercury in stack gas samples are summarized in Table 2-11.

#### 2.8.2.4 Total Chlorides (HCl/Cl<sub>2</sub>) in Stack Gas Samples

Impinger samples from the Methods 5/26A sampling train will be analyzed by ion chromatography in accordance with EPA Method 9057. QA/QC procedures for these analyses are presented in Table 2-12.

#### 2.8.2.5 Particulate Matter (Gravimetric Analysis)

Gravimetric analyses will be performed on samples collected from the Methods 5/26A Particulate/HCl/Cl<sub>2</sub> train and/or the Methods 5/29 Particulate/Metals train. Weights will be obtained on the front-half acetone rinse and particulate filter using a Denver Instruments APX-100 analytical balance. Balance accuracy is checked by using Class "S" standard weights before and after tare weighings and sample determinations. Sample fractions are dried to constant weight, defined as two successive weighings at a 6 hr interval showing a weight change of less than 0.5 mg.

#### 2.8.2.6 Stack Gas - Analysis for PCDDs/PCDFs and Other Target SVOCs

Stack flue gas samples collected using the Method 0023A/0010 sampling train will be analyzed for polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans (PCDDs/PCDFs), total mono-deca polychlorinated biphenyls (PCBs), and other semivolatile organic compounds (SVOCs), including the POHC, HCE.

Each sampling train will be prepared and split appropriately for the designated analyses. A combined front half and back half analysis will be performed for the speciated SVOCs and PCBs in order to provide the lowest detection limits. The PCDDs/PCDFs will be analyzed as separate front and back halves in accordance with Method 0023A. A schematic of the analytical scheme for the combined sampling train is provided in Appendix E.

Briefly, the XAD and filter will be spiked with internal standards for PCDDs/PCDFs, PCBs, and surrogates for the semivolatile organics and then sequentially extracted with methylene chloride and toluene. The methylene chloride and acetone rinses will be combined and added to the methylene chloride Soxhlet extract. This combined sample will be concentrated and split. The portions allocated for PCB and SVOC analyses will be combined with the acid/base neutral extracts of the impinger contents. The portion allocated for PCDD/PCDF analysis will be combined with an appropriate fraction of the toluene extract.

Method 0023A analyses (which include high resolution GC/MS as per EPA Method 8290) incorporate five isotopically labeled PCDD and PCDF field surrogates and nine labeled PCDD/PCDF internal standards. The field surrogates are spiked into the XAD resin prior to field sampling; their recoveries are monitored to assess overall method accuracy and precision. The internal standards are added to the combined XAD/filter/rinse concentrate sample at a level of 2,000 pg/sample prior to Soxhlet extraction. These internal standards are used for direct quantification of all surrogate and native PCDD/PCDF species. The addition of these standards prior to the extraction and cleanup procedures permits internal correction for any losses of target analytes that might occur during the preparation steps.

Method 8290 details instrument tune, GC column performance, and instrument calibration requirements for the analysis of stack gas samples by high resolution gas chromatography/high resolution mass spectrometry (GC/MS). Instrument calibration will be performed for all 15 2,3,7,8- substituted PCDD and PCDF isomers; data will be reported for each of these target analytes and for the total dioxins and total furans at each level of chlorination from Cl4 through Cl8.

Analysis for target PCBs will be performed by GC/MS following EPA Method 1668A (modified). At the same point when the PCDD/PCDF internal standards are added, nine isotopically labeled PCBs will be added to the combined XAD/filter/rinse concentrate sample prior to extraction. These will be used to quantify total mono-deca PCB analytes.

Analysis for semivolatile organics will be performed by low resolution mass spectrometry following the analytical protocol of SW-846, Method 8270C. Surrogates will be added prior to extraction to monitor analytical accuracy. Instrument tune and calibration procedures of these methods will apply. If lower analytical detection limits are required for HCE to provide quantitative DRE results, selective ion monitoring analysis may be employed.

QA/QC requirements for these analyses are summarized in Tables 2-13 through 2-15.

## **2.9 Internal QA Program**

Quality control checks will be performed to ensure the collection of representative samples and the generation of valid analytical results for these samples. These checks will be performed by project participants throughout the program under the direction of the APT Project Manager.

### **2.9.1 Data Collection and Sampling QC Procedures**

QC checks for the process data collection and sampling aspects of this program will include, but not be limited to, the following:

1. Use of standardized data sheets, checklists and field notebooks to ensure completeness, traceability, and comparability of the process information and samples collected.
2. Field checking of standardized forms by the APT Project Manager and a second person to ensure accuracy and completeness.
3. Strict adherence to the sample traceability procedures.
4. Submission of field biased blanks.
5. Leak checks of sample trains before and after sample collection and during the test, when appropriate.

#### 2.9.1.1 Sampling Equipment QC Checks and Frequency

Calibration of the field sampling equipment will be performed prior to and at the conclusion of the field sampling effort. Copies of the calibration sheets will be available onsite during the field sampling program for inspection, will be kept in the project file, and will be submitted in the final report. Calibrations will be performed as described in the EPA publication "Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, Stationary Source Specific Methods;" Section 2.6.2.1 presents acceptance limits.

Leak checks of the sample trains will be conducted in accordance with the protocol called out for each method. Leak checks will be conducted prior to and at the end of sample collection and during the test run, when appropriate.

#### 2.9.1.2 Sample Collection QC Checks

In order to provide a QC check on sample handling, field blanks of reagents and collection media (deionized water, filters, impinger solutions, etc.) will be placed in appropriately cleaned and sized sample containers in the field and handled in the same way as actual field samples.

For this program, sample collection QC checks and the frequency for samples to be analyzed in the laboratory are listed below:

- One field blank VOST train (i.e., one set of blank traps exposed to conditions analogous to actual samples) for each sampling day and one trip blank for the overall program.



- Three pairs of VOST tubes collected from any EPA audit cylinder provided
- One blank Method 29 sampling train
- One blank Method 26A sampling train
- One blank Method D6784-02 sampling train
- One blank Method 0023A/Method 0010 sampling train

Additionally, blank samples of each batch of sample or recovery reagent will be collected and analyzed concurrent with the emission samples. It is expected that reagent and train blank results will not be used to correct emission sample results. If unexpected blank levels are detected, blank correction procedures may be employed, but uncorrected emission values will be reported alongside the corrected values.

#### 2.9.2 Analytical QC Procedures for Samples to be Analyzed in the Laboratory

The Quality Control program for laboratory analysis makes use of a number of different types of QC samples to document the validity of the generated data. The following types of QC samples will be used during the program.

##### 2.9.2.1 Quality Control Samples and Blanks

###### Method Blanks

Method blanks contain all the reagents used in the preparation and analysis of samples and are processed through the entire analytical scheme to assess spurious contamination arising from reagents, glassware, and other materials used in the analysis.

###### Calibration Check Samples

One of the working calibration standards which is periodically used to check that the original calibration is still valid.

###### Laboratory Control Samples (LCS) or Blank Spikes

These samples are generated from spikes prepared independently from the calibration concentrates. The LCS are used to establish that an instrument or procedure is in control. An LCS is normally carried through the entire sample preparation and analysis procedure also.

###### Surrogate Spikes

Samples requiring analysis by GC/MS are routinely surrogate spiked with a series of deuterated analogues of the components of interest. It is anticipated that these compounds would assess the behavior of actual components in individual program samples during the entire preparative and analysis scheme.

The percent recovery for each surrogate will be calculated in accordance with method-specific procedures. Any values which fall outside the target QC limits described in the applicable analytical method will be flagged. Some of these recovery values may be outside the QC limit owing to matrix interferences. The following guidelines will be used:

1. All recovery data are evaluated to determine if the QC limits are appropriate and if a problem may exist even though the limits are being achieved (e.g., one compound that is consistently barely within the lower limit).
2. Any recovery data which are outside the established limits are investigated. This evaluation will include an independent check of the calculation.
3. If any of the following conditions occur, a corrective action will be initiated to determine the cause and appropriate actions will be taken.
  - All recovery values in any one analysis are outside the established limits, where one analysis is considered to be one sample analyzed by one method,
  - Over 10 percent of the values for a given sample shipment are outside limits, or
  - One compound is outside the limits in over 10 percent of the samples.

An analysis batch is defined as a group of ten or fewer samples carried through the entire preparation and analysis procedure in one batch.

Reagents used in the laboratory are normally of analytical reagent grade or higher purity; each lot of acid or solvent used is checked for acceptability prior to laboratory use. All reagents are labeled with the date received and date opened. The quality of the laboratory deionized water is routinely checked. All glassware used in the sampling and analysis procedures will be pre-cleaned according to the method requirements. Standard laboratory practices for laboratory cleanliness, personnel training, and other general procedures are used. The results of these quality control procedures will be included in the final report.

#### 2.9.2.2 Quality Control of Sorbents

Sorbents used for the organic sampling trains are provided by the laboratory after QC verification has been performed following recommended procedures in each applicable method. Additional details on sample media preparation were provided previously in Section 2.6.2.3.

## **2.10 Data Reduction, Verification, and Reporting**

Specific QC measures will be used to ensure the generation of reliable data from sampling and analysis activities. Proper collection and organization of accurate information followed by clear and concise reporting of the data is a primary goal in all such projects.

### **2.10.1 Field Data Reduction**

Appendix C of this Test Burn Protocol presents the standardized forms that will be used to record field sampling data. The APT Project Manager and at least one other field crewmember will review the data collected from each train in its entirety in the field. Errors or discrepancies will be noted and dealt with accordingly. The APT Project Manager has the authority to institute correction actions in the field. The APT Project Manager will also be notified for resolution if the situation warrants. At a minimum, the APT Project Manager is apprised of all deviations from standard protocol. Field data reduction (checking of valid isokinetic sampling rate and other sampling parameters) is done with a laptop computer using standardized Excel spreadsheets. Appendix D provides setup and recovery schematics and a description of solutions and reagents to be used in each isokinetic sampling train required for the overall program. All sample recovery sheets will be checked for completeness.

### **2.10.2 Laboratory Analysis Data Reduction**

Analytical results will be reduced to appropriate units by the laboratory using the equations given in the applicable analytical method. Unless otherwise specified, results from the analysis of waste feed and process samples for specific target constituents will be reported in units of mg/kg or % wt. Other parameters will be reported in standard units such as g/cc, Btu/lb, etc. The final test report will include all calibration data, all raw analytical data for the field samples and QA/QC samples, extraction/preparation information, and run logs in support of third party data validation procedures.

The laboratory typically reports results from the analysis of stack flue gas samples as total mass detected for the sample submitted. For those sample fractions where liquid impinger condensate is analyzed, the laboratory will measure the total liquid volume submitted and multiply by the measured concentrations of target analytes in these samples. The laboratories will generally report data as follows:

- Volatile POHC and other volatile organics - total ng collected
- Particulate matter - total mg collected in each fraction (front-half rinse and filter)
- All metals except mercury - total µg of each metal in the front-half and back-half sample train fractions
- Mercury - total µg in each sample train fraction
- HCl/Cl<sub>2</sub> - total µg as either HCl or Cl<sub>2</sub>
- PCDDs/PCDFs - total pg collected
- SVOCs - total µg collected
- PCBs - total ng collected

Each Laboratory Services Coordinator will be responsible for reviewing all results and calculations and verifying the completeness of the data set. The laboratory reports submitted by each laboratory will include the following deliverables:

- Transmittal letter listing all samples and analyses and a case narrative identifying any difficulties associated with the analyses and any anomalous QA/QC results
- Copies of Chain of Custody Forms
- Sample Report forms with sample field and laboratory identifier, dates of sample preparation and analysis, analytical results, and detection limits
- Method Blank results
- Matrix spike and matrix spike duplicate results (as applicable)
- Replicate sample analyses (as applicable)
- Laboratory Control Sample results

Reports for organics in process streams and stack samples will include the following additional information:

- Surrogate recoveries
- Summary of initial calibrations

- Continuing calibration summaries
- Instrument tunes

### 2.10.3 Data Verification

Data verification is the process of reviewing data and accepting, qualifying or rejecting it on the basis of method-specific criteria. The independent project QAO will use validation methods and criteria appropriate to the type of data and the purpose of the measurement. Records of all data will be maintained, even those judged to be “outlying” or spurious.

Field sampling data will be validated by the Field Team Leader based on a judgment of the representativeness of the sample, maintenance and cleanliness of sampling equipment, and the adherence to an approved, written sample collection procedure.

Analytical data will be validated by the subcontractor laboratory QC or supervisory personnel using criteria outlined in their laboratory-specific QA Plan and/or written SOPs. Results from field and laboratory method blanks, replicate samples, and internal QC samples will be used to further validate analytical results. Analytical results on field blanks and replicate field samples are also valuable for validation of sample collection. QC personnel will review all subcontractor laboratory raw analytical data to verify presented calculated results.

The following criteria will be used to evaluate the field sampling data:

- Use of approved test procedures
- Proper operation of the process being tested
- Use of properly operating and calibrated equipment
- Leak checks conducted before and after tests
- Use of reagents that have conformed to QC specified criteria
- Use of NIST traceable CEM calibration gases (as applicable)
- Proper chain of custody maintained

All sample trains—check to ensure proper sample gas volume collected. The criteria listed below will be used to evaluate the analytical data:

- Use of approved analytical procedures
- Use of properly operating and calibrated instrumentation
- Precision and accuracy achieved should be comparable to that achieved in previous analytical programs and consistent with objectives stated in this document.

#### 2.10.4 Final Data Reporting

Stack gas concentrations for each applicable parameter will be calculated from laboratory results and field sampling data. The total weight of the analyte detected will be divided by the volume of gas sampled to provide emission concentrations. For demonstrating MACT compliance, all emission concentrations are further corrected to 7% oxygen for comparison to the published standards.

A complete Final Report outlining the goals, methods, and results for the program will be prepared and any deviations from this test plan will be documented. The Final Report will include a section on evaluation and discussion of QA/QC results. Results will be compared to expected limits for accuracy, precision, and/or completeness as targeted in this protocol. The final test report will also include the results of any internal audits conducted on the program as well as:

- All field data sheets showing sampling method, dates, run times, personnel, equipment, and sample preservation, identification and compositing records.
- Field equipment calibration data.
- Analytical lab reports and relevant supporting documentation.

The final report will be reviewed by an independent 3<sup>rd</sup> party. The results of the independent audit will be contained in a separate report.

#### **2.11 Routine Maintenance Procedures and Schedules**

This section provides pertinent information for field sampling equipment as well as a listing of all critical facility equipment necessary to maintain permitted operating conditions and to demonstrate continuing permit compliance. Information is provided for preventive maintenance and schedules and spare parts for key equipment and instrumentation.

### 2.11.1 Field Sampling Equipment

The field team follows an orderly program of positive actions to prevent the failure of equipment or instruments during use. This preventive maintenance and careful calibration helps to ensure accurate measurements and minimal field delays.

All equipment that is scheduled for field use is calibrated as outlined previously in Section 2.6.2.1. Prior to each field use for a specific project, the equipment is cleaned and checked to ensure it is in good working order. An adequate supply of spare parts and sample train glassware is brought to each site to minimize downtime and field sampling delays. Any equipment that does experience problems is appropriately tagged in the field to ensure that it is repaired upon return to the office.

### 2.11.2 Facility Equipment and Instrumentation

During scheduled shutdowns, all waste feed lances are pulled and inspected. If necessary, the lance is repaired, otherwise it is replaced. All seals and expansion joints from the feed end to the stack are inspected and repaired as needed. Instrumentation is maintained and calibrated as required by the facility's RCRA permit:

- Attachment 13 – Instrument Calibration,
- Attachment 15 – QAPP for CO<sub>2</sub> and O<sub>2</sub> continuous emission monitors, and
- Attachment 16 – PI Archiving System.

## **2.12 QA/QC Assessment Procedures**

The QA activities implemented in this program will provide a basis for assessing the accuracy and precision of the analytical measurements. Section 2.9 of this TBP discusses the QA activity that will generate the accuracy and precision data for each sample type. A generalized form of the equations that will be used to calculate accuracy, precision, and completeness follows.

### 2.12.1 Accuracy

Percent accuracy will be determined using the following equation:

$$\% \text{ Recovery} = \frac{(X - S)}{T} \times 100$$

where:

X = experimentally determined concentration of the spiked sample  
T = true concentration of the spike  
S = sample concentration before spiking

### 2.12.2 Precision

Precision will be determined using the following equation:

$$\text{Relative Percent Difference (RPD)} = \left[ \frac{(D_1 - D_2)}{\left\{ \frac{D_1 + D_2}{2} \right\}} \right] \times 100$$

where:

D<sub>1</sub> and D<sub>2</sub> = results of duplicate measurements or standard deviation relative to the average value expressed as relative standard deviation:

Relative standard deviation will be expressed as follows:

$$\text{Relative Standard Deviation (\% RSD)} = \left\{ \frac{\sigma_{(n-1)}}{\bar{X}(x_1 \dots x_n)} \right\} \times 100$$

where:

σ (n-1) = standard deviation of the sample data

n = number of replicates

$\bar{X}(x_1 \dots x_n)$  = arithmetic mean of the sample data

### 2.12.3 Completeness

Data completeness is a measure of the extent to which the database resulting from a measurement effort fulfills objectives for the amount of data required. For this program, completeness will be defined as the percentage of valid data for the total valid tests. Completeness is assessed using the following equation:



$$Completeness (\%) = \left[ \frac{D_r}{D_c} \right] \times 100$$

where:

$D_r$  = number of samples for which valid results are reported

$D_c$  = number of valid samples that are collected and reach the laboratory for analysis

The completeness objective will help to evaluate the accuracy and precision of the analytical measurements.

### **2.13 External QA Program**

The External Quality Assurance Program includes both performance and system audits as independent checks on the quality of data obtained from sampling, analysis, and data gathering activities. Every effort is made to have the audit assess the measurement process in normal operation. Either type of audit may show the need for corrective action.

#### **2.13.1 Performance Audits**

The sampling, analysis, and data handling segments of a project are checked in performance audits. A different operator/analyst prepares and conducts these audit operations to ensure the independence of the quantitative results.

EPA Quality Control concentrates or other standards will be used to assess the analytical work. Results will be reviewed by the subcontractor laboratory and QC personnel. Any additional audit samples presented by the regulatory agencies will be analyzed, along with program samples, by the appropriate lab and at the same time as all other samples. It will, however, be the responsibility of the regulatory agency to obtain these samples and present them to the facility project manager in a form that is amenable and appropriate to the analytical methods being utilized.

If the regulatory agency advises the facility program manager that audit results fall outside of acceptable ranges, the analytical data will be further reviewed for error in conjunction with the agency. If a simple, correctable error is found (e.g., an arithmetic error), correction will be made and results resubmitted. If no simple error is found, an investigation into other causes of the failure (e.g., lack of sample integrity) will be conducted and the results will be evaluated in terms of their impact on sample data integrity.

#### **2.13.2 Corrective Action**

The acceptance limits for the sampling and analyses to be conducted in this program will be those stated in the method or defined by the project manager. The corrective actions are likely to be immediate in nature and most often will be implemented by the analyst or Project Manager; the corrective action will usually involve recalculation, reanalysis, or repeating a sample run. Ongoing corrective action policy is described here.

#### 2.13.2.1 Immediate Corrective Action

Specific QC procedures and checklists are designed to help analysts detect the need for corrective action. Often the person's experience will be more valuable in alerting the operator to suspicious data or malfunctioning equipment.

If a corrective action can be taken at this point, as part of normal operating procedures, the collection of poor quality data can be avoided. Instrument and equipment malfunctions are amenable to this type of action and QC procedures include troubleshooting guides and corrective action suggestions. The actions taken should be noted in field or laboratory notebooks, but no other formal documentation is required, unless further corrective action is necessary. These on the spot corrective actions are an everyday part of the QA/QC system.

Corrective action during the field sampling portion of a program is most often a result of equipment failure or an operator oversight and may require repeating a run. When equipment is discovered to be defective (i.e., pre- and post-sampling leak check), it is repaired or replaced and a correction factor is established as per the EPA method. If a correction factor is unacceptable, the run is repeated. Operator oversight is best avoided by having field crew members audit each other's work before and after a test. Every effort is made by the field team leader to ensure that all QC procedures are followed. Economically, it is preferred to repeat a run during a particular field trip rather than return at a later date.

Corrective action for analytical work would include recalibration of instruments, reanalysis of known QC samples and, if necessary, of actual field samples.

If the problem is not solved in this way, more formalized, long-term corrective action may be necessary.

#### 2.13.2.2 Long-Term Corrective Action

The need for this action may be identified by standard QC procedures, control charts, performance, or system audits. Any quality problem which cannot be solved by immediate corrective action falls into the long-term category. The condition is reported to a person responsible for correcting it and who is part of the closed loop action and follow up plan.

The essential steps in the closed loop corrective action system are:

- Identify and define the problem.
- Assign responsibility for investigating the problem.
- Investigate and determine the cause of the problem.
- Determine a corrective action to eliminate the problem.
- Assign and accept responsibility for implementing the corrective action.
- Establish effectiveness of the corrective action and implement it.
- Verify that the corrective action has eliminated the problem.

Documentation of the problem is important to the system. A Corrective Action Request Form is filled out by the person finding the quality problem. This form identifies the problem, possible causes, and the person responsible for action on the problem. The responsible person may be an analyst, field team leader, department QC coordinator, or the APT Technical Director. If no person is identified as responsible for action, the APT Technical Director investigates the situation and determines who is responsible in each case.

The Corrective Action Request Form includes a description of the corrective action planned and the date it was taken, and space for follow up. The APT Technical Director checks to be sure that initial action has been taken and appears effective and, at an appropriate later date, checks again to see if the problem has been fully solved. The APT Technical Director receives a copy of all Corrective Action Forms and then enters them in the Corrective Action Log. This permanent record aids the APT Technical Director in follow up and makes any quality problems visible to management; the log may also prove valuable in listing a similar problem and its solution.

### 2.13.3 Quality Assurance Reports to Management

#### 2.13.3.1 Internal Reports

The Laboratory Services Coordinator will prepare a written report on QC activities associated with this project for the APT Technical Director. This report will detail the results of quality control procedures, problems encountered, and any corrective action which may have been required.

All Corrective Action Forms are submitted to the APT Technical Director for initial approval of the corrective action planned and a copy is provided to the CHA Project Manager.

#### 2.13.3.2 Reports to Client

The final report will include a section summarizing QA/QC activities during the program. The Project Manager, Laboratory Services Coordinators and the APT Technical Director will participate in preparing this section. This section will provide summary QA/QC results for method blanks, surrogate spikes and laboratory control spike recoveries. This section will evaluate overall data quality in terms of accuracy, precision, and completeness. Any discrepancies or difficulties noted in program work, protocol deviations, or documentation gaps will be identified and discussed.

**Table 2-1 – Laboratories Performing Analyses**

<b>Parameter</b>	<b>Stream <sup>a</sup></b>	<b>Laboratory <sup>b</sup></b>
Viscosity, Total Chlorine, Heat Content and Ash Content	Waste	Aragonite
Hexachloroethane	Waste Liquids	Aragonite
Monochlorobenzene	Waste Liquids	Aragonite
PCBs	Waste Liquids, Process	Aragonite
Metals	Waste, Process	Aragonite
Semivolatile Organics, Volatile Organics, Herbicides, Pesticides, and Cyanide	Process	Aragonite
Particulate Matter	Scrubber Outlet and/or Incinerator Stack	APT
Hydrogen Chloride / Chlorine	Scrubber Outlet and/or Incinerator Stack	STL – W. Sac
Metals	Scrubber Outlet and/or Incinerator Stack	STL – W. Sac
Mercury	Scrubber Outlet and/or Incinerator Stack	STL – W. Sac
Volatile Organics	Scrubber Outlet and/or Incinerator Stack	STL – Knoxville
PCDDs/PCDFs	Scrubber Outlet and/or Incinerator Stack	STL – W. Sac
PCBs and Semivolatile Organics	Scrubber Outlet and/or Incinerator Stack	STL – W. Sac
<sup>a</sup> Waste Feed Streams include containerized solids, apron feed solids, sludge materials, direct burn liquid waste (energetic), aqueous liquids and liquid blend. Process streams include kiln slag and combined spray dryer solids / baghouse solids.  <sup>b</sup> Aragonite = Clean Harbors Onsite Laboratory or Utah-certified outside laboratory supervised by Clean Harbors laboratory personnel APT = Air Pollution Testing's Laboratory – Arvada, Colorado STL - W. Sac = Severn Trent Laboratories, West Sacramento, CA STL - Knoxville = Severn Trent Laboratories, Knoxville, TN		

**Table 2-2 – Waste Feed Parameters and Methods**

Parameter	Method
Total chlorine (preparation)	EPA M 5050
Total chlorine (analysis)	EPA M 9056
Viscosity (liquids only)	ASTM D 2983
Heat content	ASTM D 240

**Table 2-3 – Sampling and Analytical Program Summary for Waste Feed and Process Streams**

Waste Feed and Process Streams	Sampling Method (c)	Sampling Frequency	Analytical Parameters	Analytical Method	Total Samples for Analysis		
					Total Field	Lab QC	Total
Waste Solids (a)	Scoop	(d) (e)	Total Chlorides	EPA M 5050 / 9056	6	2	8
			Heat Content	ASTM D 240	6	2	8
			Metals	EPA M 6010B/7470A/7471A	6	4	10
Pumpable Wastes (b)	Tap	(e)	Viscosity	ASTM D 2983	48	16	64
			Total Chlorides	EPA M 5050 / 9056	48	16	64
			Heat Content	ASTM D 240	48	16	64
			Metals	EPA M 6010B/7470A/7471A	48	32	80
			PCBs	EPA M 8082	48	16	64
Fuel Oil	Tap	(f)	Viscosity	ASTM D 2983	1	1	2
			Total Chlorides	EPA M 5050 / 9056	1	1	2
			Heat Content	ASTM D 240	1	1	2
Spiking Solutions	Coliwasa	(g)	Chromium	EPA M 6010B	25	3	28
			Mercury	EPA M 7470A	4	1	5
Slag and Baghouse/ Spray Dryer Residue	Scoop	(h)	Metals	(i)	4	1	5
			VOCs	EPA M 8260B	4	1	5
			SVOCs	EPA M 8270C	4	1	5
			Herbicides	EPA M 8151	4	1	5
			Pesticides	EPA M 8081	4	1	5
			Cyanide	EPA M 9010A	4	1	5
			PCBs	EPA M 8082	4	1	5

**(a)** Waste solids (2 streams) include containerized solids and apron feed solids.

**(b)** Pumpable wastes (8 streams) include sludge, direct feed, aqueous waste and liquid blend fed to the kiln, North ABC liquid blend and aqueous wastes, and South ABC liquid blend and aqueous wastes.

(c) Sampling method designations from EPA-600/8-84-002, February 1984.

(d) Containers will be prepared and characterized prior to the Test Burn as per the facility's WAP. The total samples for analysis column refers to apron feed solids. One composite will be analyzed for each Test Run.

(e) One grab sample for each waste stream every 15 minutes; one composite sample for each waste stream per run, and one duplicate per waste stream

(f) One grab sample will be collected from the feed tank.

(g) Each drum of chromium acetate and mercuric acid will be sampled.

(h) One composite sample collected per day of testing as per facility's WAP. The number of samples is based on four days of testing.

**(i) EPA Methods 1311 (TCLP), 3050B or 3051, 6010B, 7470A and 7471A.**

Sampling Method	Analytical Parameters	Analytical Method	Total Samples Analyzed (see Note)				Total (e)
			Condition 1		Condition 2		
			Field Samples	Train and Reagent Blanks	Field Samples	Train and Reagent Blanks	
EPA M 5 / 26A	PM	EPA M 5	3	1 TB, 1 RB	3	--	8
	HCl and Cl <sub>2</sub>	EPA M 9057	3	1 TB, 1 RB	3	--	8
EPA M 0023A	PCDDs and PCDFs	EPA M 23 / M 8290	3	1 TB, 1 RB	3	0	8
EPA M 0010	PCBs (a)	EPA M 1668A	3	1 TB, 1 RB	--	--	5
EPA M 0010	SVOCs (b)	EPA M 8270C	3	1 TB, 1 RB	--	--	5
EPA M 29	Metals (c)	EPA M 6010B/6020	3	1 TB, 1 RB	3	0	8
ASTM D6784-02	Mercury	EPA M 7470A	3	1 TB, 1 RB	3	0	8
EPA M 0030 (VOST) Tubes	VOCs (d)	EPA M 5041A / 8260B	3 runs (18 tubes)	3 Field Blanks, 1 Trip Blank	--	--	22
VOST Tube Prep		EPA M 5041A	30	0	--	--	30
VOST Condensate	VOCs (d)	EPA M 8260B	3 runs	1 Field Blank	--	--	4
Facility CEM	O <sub>2</sub> , CO <sub>2</sub> , CO, NO <sub>x</sub> , SO <sub>2</sub> , THC	Facility CEM QA Plan	3	0	3	0	6

(a) Target PCBs will include total mono-deca congeners.

(b) Target SVOCs: organic compounds identified in Method 8270C (including Hexachloroethane).

(c) Target Metals include : Pb, Cd, As, Be, Cr, Se, Sb, Ba, Co, Cu, Mn, Ni, Ag, and Tl.

(d) Target VOCs: organic compounds identified in Method 8260B (including Monochlorobenzene).  
VOST tube analysis assumes that all pairs from each run are analyzed individually.

(e) Audit samples will be analyzed for any methods if provided by the various regulatory agencies.

**Note:**  
Metals, PCDD and PCDF samples will be analyzed as separate front and back halves.  
PCB and SVOC samples will be analyzed as a single sample per run, which will include the condensate.  
VOST samples will be analyzed as 6 separate tubes and 1 condensate per run.  
Mercury samples will be analyzed as 3 fractions per run (particle bound, oxidized and elemental).



**Table 2-5 – Sample Preservation and Holding Time Requirements, Stack Gas Samples**

Parameter	Matrix	Preservation	Holding Time (a)
Volatile Organics (Method 0030)	Aqueous	4°C	14 days
	Tenax and Tenax/charcoal	4°C	14 days
PCDDs/PCDFs, PCBs and Other SVOCs (Method 0023A/0010)	XAD Resin	4°C	14 days (to extraction – SVOC requirement)
			40 days (extraction to analysis – SVOC requirement)
Chloride/Chlorine (Method 26A)	Aqueous	N/A	30 days
Metals (Method 29) (except Hg)	Aqueous	N/A	6 months
	Solid / Filter	N/A	6 months
Mercury (ASTM 6784-02)	Aqueous	No temperature requirement, various solution stabilization procedures – see Method for details	28 days
	Solid/Filter	N/A	28 days
<b>(a) Holding times will be calculated from the day of sample collection.</b>			

**Table 2-6 – Sample Preservation and Holding Time Requirements, Waste Feed and Process Stream Samples**

Parameter	Preservation	Holding Time
Metals	4 °C	6 months
Metals – Mercury	4 °C	28 days
Total Chlorides	4 °C	30 days
SVOCs, PCBs, Herbicides and Pesticides	4 °C	14 days to extraction; 40 days extraction to analysis
MCB	4 °C	14 days
Cyanide	4 °C	14 days to extraction; immediate analysis following extraction or keep sample at 4°C

**Table 2-7 – Summary of QA/QC Procedures for Chlorine in Waste Feed**

Quality Parameter	Method Determination	Frequency	Target Criteria
Calibration	Initial analysis standards	Prior to sample analysis	Instrument dependent
	Continuing calibration standards	Before and after sample analysis; once per batch	90%-110% of expected value
Accuracy - calibration	Analysis of calibration check standard	After every calibration	90%-110% of expected value
Accuracy	Reference material	Once per batch	90% to 110% of expected value
Precision	Duplicate preparation and analysis of at least one run's samples	Once per waste stream	10% RPD
Blank	Method blank carried through all sample preparation and analysis steps	Once per batch	Below detection limit
RPD – Relative Percent Difference			

**Table 2-8 – Summary of QA/QC Procedures for Metals in Waste Feed**

Quality Parameter	Method Determination	Frequency	Target Criteria
Calibration	Initial analysis of standards at different concentration levels	At least once before sample analysis	Instrument-dependent. Linear corr. coefficient of std. Data $\geq 0.995$
	Continuing mid-range calibration standard	Before and after sample analysis	80% to 120% of expected value for GFAA. 90% to 110% of expected value for ICAP.
Interference check	Interference check sample	Before and after ICAP analysis	80% to 120% of expected value
Accuracy – calibration	Analysis of calibration check standard	After every initial calibration	90% to 110% of expected value
Accuracy	Aliquot of one sample from a run spiked with analytes at 3 times the detection limit or twice the sample level	One per sample matrix	70% to 130% recovery
Precision	Duplicate preparation and analysis of one sample from each matrix	One per sample matrix	Range < 35% if sample result above lowest standard
Blank	Method blank carried through all sample preparation and analysis steps	Once per sample batch	Below detection limit
GFAA = graphite furnace atomic absorption CVAA = cold vapor atomic absorption ICAP = inductively coupled argon plasma			

**Table 2-9 – Summary of QA/QC Procedures for POHCs in Waste Feed**

Analytical Parameter	QC parameter	Method of Determination	Frequency	Objective
PCBs (Method 8082)	Accuracy	Surrogate Recovery for Decachlorobiphenyl Tetrachloro-m-xylene	Every sample	50-130%
	Accuracy	Matrix spike (Aroclor 1016 and Aroclor 1260)(a)	One per stream	50-130%
	Precision	Surrogate Recovery for Decachlorobiphenyl Tetrachloro-m-xylene	Calculate RSD for each stream	< 35% RSD
	Precision	Duplicate preparation and analysis of one sample from each matrix	One per stream	< 35% RPD
(a) Matrix spikes not applicable for samples with > 0.1% of the target analyte (i.e., liquid blend material). Replicate analysis will be done on any such sample with the control limit determined by the Aragonite laboratory.				

**Table 2-10 – QA Objectives for VOST Analyses**

Quality Parameter	Method Determination	Frequency	Target Criteria
Accuracy	Initial Calibration – 5 point	Prior to analysis	RRF of CCC: %RSD +/- 30%  Minimum RF for SPCCs: > or = 0.100 (chloromethane, 1,1-dichloroethane, bromoform) > or = 0.300 (1,1,2,2-tetrachloroethane, chlorobenzene)
	Continuing calibration	every 12 hours	1 point on initial curve
Blanks – sample integrity and field contamination	Field blanks, 1 pair of traps	One pair per run	Less than RL
Blanks – verify no contamination from storage / shipment	Trip blanks, 1 pair of traps	One pair per shipment	Less than RL
Blanks – verify no lab contamination and system control	Lab blanks, 1 pair of traps	Daily, before analysis of samples and in-between high-level samples	Less than RL, Except for common laboratory solvents
Consistency in chromatography	Monitor internal standard; retention time and area	Every sample, standard and blank	Retention time within $\pm 30$ sec of last calibration check; area within -50 to +200%D
Accuracy	Surrogate recovery for Toluene-d8 (a)	Every sample	50% - 150% recovery
Verification of VOST system accuracy	Analysis of samples from EPA audit cylinder, if provided	Once per test	Within 50% - 150% of certified concentration
Precision	Surrogate recovery for Toluene-d8	Calculate overall RSD	< 35% RSD
Breakthrough determination	Separate analysis of front and back traps	All sample runs Unnecessary for blanks	Quantity on TX/C must be < 30% of amount on TX trap - does not apply when < 75 ng on TX/C trap
RRF – Relative Response Factor CCC – Calibration Check Compound SPCC – System Performance Check Compound RSD - Relative Standard Deviation RL – Reporting Limit (a) – All surrogate recoveries will be reviewed – Toluene-d8 will be most pertinent for evaluation of the POHC.			

**Table 2-11 – QA Requirements for Metals in Stack Gas**

Quality Parameter	Method Determination	Frequency	Target Criteria
<b><u>Metals Other Than Mercury (ICP-MS)</u></b>			
Calibration	Initial analysis of standards at multiple levels	At least once	Method-dependent. Linear correlation coefficient of standard data > 0.995
	Continuing mid-range calibration standard	Every 10 samples and at end of day	90-110% for ICP-MS
	Continuing calibration blank	With continuing calib. std.	Below RL
Accuracy – calib.	Analysis of calibration check standard	After every initial calib.	90% to 110% of true value
Accuracy	Matrix spike (post-digestion spikes)	Once per test	70% to 130% recovery
Precision	MS / MSD (post-digestion spikes)	Once per test	RPD < or = 35%
Blanks	Field Blanks & Method Blanks	One each per test	Below RL
<b><u>Mercury (CVAAS)</u></b>			
Calibration	6-point : Blank and 5 standards, lowest is reporting limit (RL)	Daily	R <sup>2</sup> >0.995
	Continuing Calibration Verification (CCV) – may also be LCS	Every 10 samples and at end of day	+/- 20% of true value
Accuracy (calibration)	Second Source Initial Calibration Verification (ICV)	Daily	+/- 10% of true value
Accuracy	Matrix spikes (MS)	Once per test, Nitric/Peroxide fraction	80% - 120% recovery
	Laboratory control samples (LCS)	Once per test, all fractions	80% - 120% recovery
Precision	Matrix spike duplicate (MSD)	Once per test, Nitric/Peroxide fraction	RPD < or = 35%
Contamination	Method blanks (MB)	Once per test	Below RL
	Field blanks	Once per location	Below RL
	Reagent blanks	Once per batch	
RPD - Relative Percent Difference ICP-MS = Inductively Coupled Argon Plasma Mass Spectroscopy RL – Reporting Limit			

**Table 2-12 – QA Requirements for Chlorides in Stack Gas**

Quality Parameter	Method Determination	Frequency	Target Criteria
Calibration (qualitative)	Relative retention time	Every calibration curve	±3 standard deviations of average
	Average retention time	Every calibration curve	Within retention time window of stds.
Calibration (quantitative)	Initial calibration with a minimum of four standards	At least once before sample analysis	Linear correlation coefficient > 0.995
	Continuing calibration	Every 10 samples and at end of day	90% - 110% of theoretical conc.
Accuracy (calibration)	Certified reference solution	After every initial calibration and before sample analysis	90% - 110% of true value
Accuracy	Matrix spikes	Once per test	85% - 115% recovery
Precision	MS/MSD	Once per test	< 25%
	Duplicate analysis	All emissions samples	< 5% RPD or additional duplicate analysis and use of all four values
Contamination	One method blank carried through sample preparation and analysis	Once per test	Below RL
	Field blanks	Once per location	
	Reagent blanks	Once per batch	
RPD = Relative Percent Difference			

**Table 2-13 – QA Objectives for PCDD/PCDF Analysis of Stack Gas Samples**

Quality Parameter	Method Determination	Frequency	Target Criteria
Calibration	Five-level calibration curve; continuing calibration standard	At least once; continuing calibration check at beginning of each 12-hr shift	<u>Initial:</u> ≤20% RSD for unlabelled standards ≤30% RSD for internal standards S/N ratio ≥2.5; Isotope ratios within control limits <u>Continuing:</u> ≤20% of ICAL for 17 unlabelled stds ≤30% of ICAL for internal standards S/N ratio ≥2.5; Isotope ratios within control limits
Continuing Calibration Check	Analysis of calibration check	Daily	80% - 120% of theoretical value
Accuracy-surrogates	Spiked into samples prior to sampling	Every sample	70% - 130% recovery
Accuracy-internal standards	Spiked into samples prior to extraction and analysis	Every sample	40%-130% recovery for tetra – hexa 25%-130% for hepta & octa homologs
Accuracy – audit samples	Prepared and analyzed along with program samples	Presented by the regulatory agency	Determined by regulatory agency
Blanks	Method blank for each component	One per batch of samples	ND or <5% of field concentration
	Field Blank	Once per test	
Mass Spectrometer Performance	Section 8.2.2 of Method 8290	At beginning of each 12-hr period	Static resolving power of 10,000 (10% valley definition)
Qualitative Identification	Retention Time and GC Column Performance	Every sample	Compliance with Section 8.2.1 of Method 8290
S/N = Signal to Noise Ratio RSD = Relative Standard Deviation ICAL = Initial Calibration			



**Table 2-14 – QA Objectives for SVOC Analysis of Stack Gas Samples**

Quality Parameter	Method Determination	Frequency	Target Criteria
Accuracy (calibration, internal standards)	Initial Calibration – five point	As needed, see CCC	System performance check compound relative response factors >0.050
	Continuing Calibration Check (CCC)	Every 12 hours	RSD < or = 20%
	Internal Standards	Every sample	Relative response time +/- 30 seconds Accuracy 50% - 200%
Accuracy (surrogates)	1,2-dichlorobenzene-d4 spiked into samples prior to sampling and/or analysis	Every sample	See Appendix B <sup>(a)</sup>
Accuracy	LCS - Representative SVOCs	Once per Extraction Batch	See Appendix B <sup>(a)</sup>
Precision	LCSD – Representative SVOCs		See Appendix B <sup>(a)</sup>
Method Blank	Each target analyte	Once per batch	Blank value < RL
Field Blank	Each target analyte	Once per test	Below RL
RSD = relative standard deviation RPD = relative percent difference  (a) – Note that Appendix B historical values may be updated as necessary for the current program. Conformance with required MDL on HCE by SIM will be demonstrated with a low-level calibration standard.			

**Table 2-15 – QA Objectives for PCB Analysis of Stack Gas Samples**

Quality Parameter	Method Determination	Frequency	Target Criteria
Accuracy Accuracy	Initial Calibration - Five point calibration	initially and as required	targets (natives) 40% RSD
	Continuing Calibration - Midpoint standard	every 12 hours	native response factors within 40% of initial calibration, ion ratios within 15% of theoretical
Accuracy-surrogates	Isotopically-labelled compounds spiked into samples prior to sampling and/or analysis	Every sample	50% - 150% recovery
Precision-surrogates	Same as for accuracy-surrogates; pool results for each PCB component	Every sample	< 50% RSD
Method Blank	Each PCB analyte	Once per batch	Blank value < RL
Field Blank	Each PCB analyte	Once per test	Below RL
RSD = relative standard deviation			

